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CHAPTER 1

INTRODUCTION

The cold rolling industry makes quality sheet products for aerospace and packaging applications [1]. The aluminium cold rolling industry has used mineral oil-based lubricants (MB) for a time because mineral oil-based lubricants (MB) give good wear protection and cost less. Mineral oil-based lubricants (MB) exhibit low biodegradability and tend to persist in the environment for extended periods. Due to their stable chemical structure, they do not break down easily under natural conditions, leading to accumulation in soil and water bodies. When mineral oil-based lubricants (MB) are disposed of, they create problems and attract regulatory review. The aluminium cold rolling industry now needs alternatives for these lubricants [2]. It has been noted that recent advancements in lubricants indicate that palm oil-based emulsions are promising for metal-forming processes. The palm oil-based emulsions are **bio-based lubricants** (BB) that **have benefits such as** renewability, **non-**toxicity **and** biodegradability. The benefits match global sustainability goals. However, the performance of palm oil-based emulsions (BB) may does not always equal efficiency that of conventional lubricants [3]. This study looks at a missing piece, in aluminium cold rolling. It tests how BB lubricants perform technically and how they affect the environment under certain conditions. The main idea is that BB lubricants can provide friction performance comparable to that of MB lubricants while offering environmental benefits. In this study aluminium alloy strips, were cold rolled to see how BB lubricants work at reduction ratios from 10 per cent to 40 per cent and at rolling speeds from 5 meters per minute to 20 meters per minute. The experimental framework combines friction testing, mechanical property testing, and environmental impact testing. The experimental framework allows us to compare BB and MB easily. Earlier research looked at lubricants in machining and light metal forming. Earlier research did not look much at lubricants in high-strain processes like rolling [4,5]. Study

[6,7] showed that vegetable-oil-based emulsions lowered the friction and these vegetable-oil-based emulsions improved smoothness in aluminium processing.

The key contributions of this work are three. First the work makes a direct performance comparison between BB and MB lubricants. The work quantifies friction coefficients, rolling forces and surface quality, under different rolling conditions. Second the work evaluates the benefits of BB. The work includes BB biodegradability and BB chemical oxygen demand to underscore BB superiority. Third point out the trade-offs, such, as cost and power consumption that help adoption. It has analysed insights connect lab findings with world use and give a plan for lubrication in aluminium rolling.

1.1 Rolling Process

In most cases, rolling is the initial step in turning raw materials into a finished good. Cold-rolling sheet and strip further strengthens the material, making it thinner or even turning it into foil, among other uses.

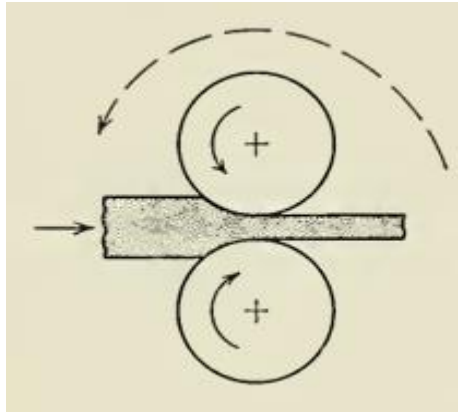


Fig. 1.1 Basic rolling process

From a weight viewpoint, rolling is clearly predominant among all metal forming processes, accounting for the majority of metallic products manufactured worldwide. Standard and superior-quality products can be produced at relatively low cost by rolling due to its high productivity and dimensional accuracy [8,9].

Fig. 1.1 shows the basic rolling process for sheet and plates. Sheet and plate rolling is a metal forming process, carried out either in hot or cold conditions, in which the cross-

sectional area of the material is reduced by passing it between rotating rolls. During this process, the material undergoes simultaneous elongation and lateral spread while its thickness decreases. [10].

In the cold rolling process, metal workpiece is fed between two rolls that rotates in opposite directions, with the roll gap set smaller than the thickness of the incoming material [11]. Since the surface velocity of the rolls exceeds the velocity of the feed material, friction at the roll–metal interface provides the necessary driving force that enables the metal workpiece to be drawn into the roll gap [12]. During deformation, the metal is compressed and elongated to compensate for the reduction in cross-sectional area, ensuring volume constancy [13]. The extent of deformation, product quality, and overall efficiency of the rolling process are strongly influenced by the frictional conditions prevailing between the tribological pairs [14].

For complete control and optimization of the rolling process, several independent parameters—such as starting material, initial geometry, roll and tool geometry, lubrication conditions, temperature, deformation speed, and degree of reduction—must be systematically examined [14], [15]. These parameters directly affect dependent variables including power requirement, final mechanical properties, exit temperature, surface finish, and dimensional precision of the rolled product [16].

1.2 Tribological Properties

Tribological properties such as friction, wear, and lubrication play a critical role in ensuring an efficient rolling process [8], [9]. Friction is the resistance to sliding motion along an interface. In rolling operations, a controlled level of friction is necessary to pull the strip into the roll gap and sustain deformation [11], [12]. However, excessive friction can increase rolling forces, power consumption, and surface damage [13].

Reducing friction and wear forces and making relative motion between contact pairs easier are the goals of lubricant introduction. The COFs that are produced when two surfaces are in relative motion can be represented using a conventional Stribeck curve, as illustrated in Fig. 1.2. This curve represents the variation of friction as a function of key operating



parameters, namely the lubricant viscosity (η), sliding speed (v), and the applied contact load (F).

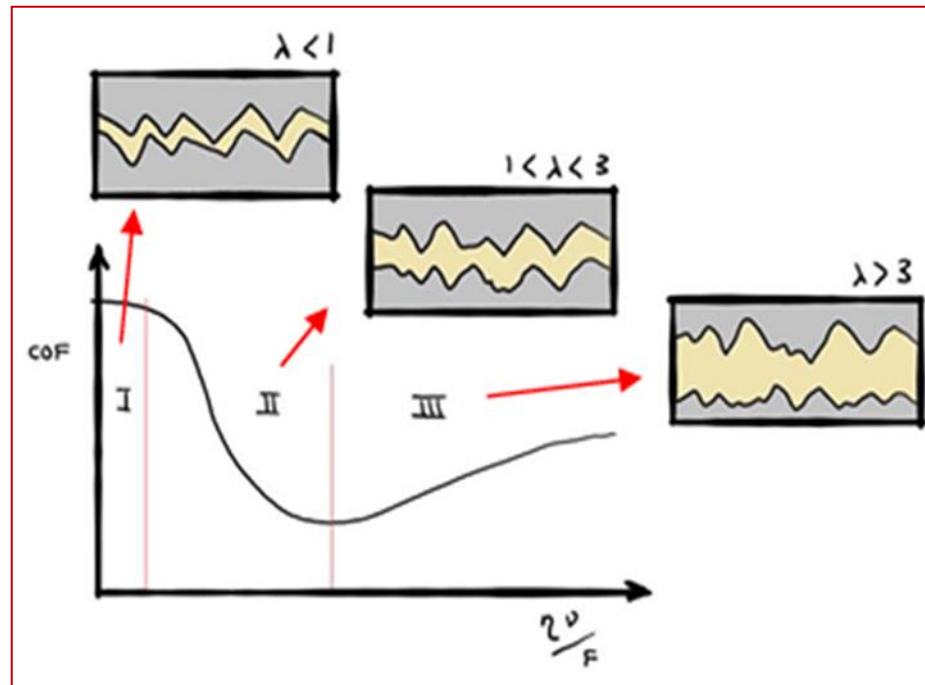


Fig. 1.2 Examining the changes in the coefficient of friction (COF) and lambda ratio (λ) as a function of lubricant viscosity η , sliding speed v , and contact force F in three different lubrication regimes: boundary, mixed, and hydrodynamic [22].

In the inset image, we can see variation across several regimes in the amount of asperity contacts between the 2 surfaces, measured as the ratio λ .

Wear behavior is another closely related concern in rolling tribology. Since the workpiece interacts with the tooling only during a single pass, wear of the workpiece surface is generally not objectionable; in fact, the shiny and fresh metal surface generated due to mild abrasive wear is often considered desirable [9], [14]. In contrast, wear of the rolling tools is highly undesirable because tooling is expensive and expected to maintain dimensional integrity over long service periods [10], [16]. Tool wear leads to dimensional inaccuracies, increased frictional resistance, higher rolling loads and power requirements, as well as production downtime associated with tool replacement and maintenance [13], [15].

Lubrication is therefore a key factor in controlling friction and wear in rolling processes. Rolling lubricants function not only as friction modifiers but also serve as coolants, thermal barriers, corrosion inhibitors, and parting agents [13], [17]. Consequently, lubricant selection is of immense importance and must consider factors such as ease of application and removal, non-toxicity, low odor, low flammability, chemical stability or controlled reactivity with material surfaces, and adaptability over an extensive range of temperatures, pressures and rolling speeds [17], [18]. Additional considerations include surface wetting characteristics, cost, availability, and the ability of the lubricant to flow or shear-thin while maintaining functional performance [14], [18].

It is important to note that lubricant performance is highly sensitive to interfacial conditions. Variations in contact area, applied load, rolling speed, temperature, and lubricant supply significantly influence frictional behavior, wear mechanisms, and lubrication regimes during rolling [11], [13], [16].

1.3 Role of Lubrication in Cold Rolling

The global industrial lubricants market, which includes machinery lubricants for applications such as engines, hydraulics, compressors, bearings, and construction equipment, was valued at USD 60,955.2 million in 2024 and is expected to witness steady growth expanding at a compound annual growth rate (CAGR) of 4.50 percent from 2024 to 2033. This expansion is driven by increasing industrialization in emerging economies like India, China, Brazil, South Africa, and Japan, alongside rising demand from sectors including chemicals, mining, unconventional energy, metalworking, and plastics production. Asia-Pacific currently holds the dominant regional share at 46.4% in 2024, underscoring the role of these markets in fueling lubricant consumption for heavy machinery and manufacturing processes Fig. 1.3 shown the data [21].

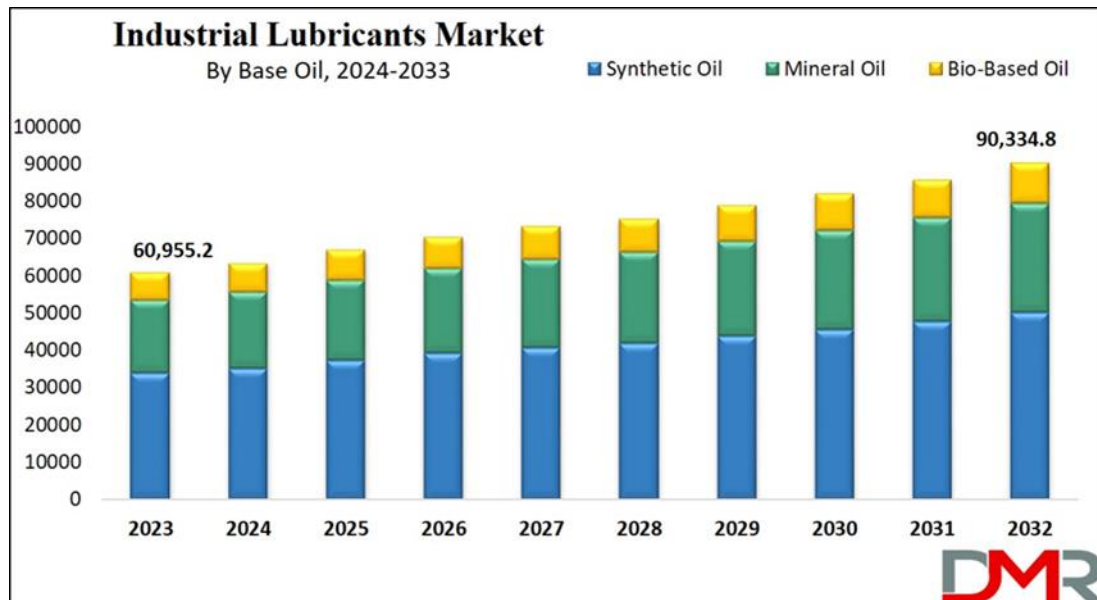


Fig. 1.3 Industrial Lubricant Market by base oil 2024-2032 [21]

1.3.1 Lubrication Regimes in Rolling

Depending on speed, viscosity, and load, the lubrication in cold rolling can span boundary, mixed, or hydrodynamic regimes, as classically described by the Stribeck curve (a plot of friction coefficient vs. a parameter like viscosity x speed/load). Fig. 1.2 shows a schematic Stribeck curve with three distinct regions [20-23]. In boundary lubrication (region 1), the lubricant film is very thin and the asperities of roll and strip are in frequent contact – friction is relatively high and governed by surface interactions and any boundary additives. In hydrodynamic lubrication (region 3), the speed and viscosity are sufficient to form a continuous fluid film separating the surfaces, resulting in low friction drag mainly due to viscous shear within the lubricant [25]. Cold rolling typically carried out in the mixed lubrication regime (region 2), where the load is shared between a thin lubricant film and direct asperity contact between the interacting surfaces [26-27]: the strip speed and lubricant viscosity are enough to generate a partial fluid film, but the extremely high contact pressure (often on the order of 500–1000 MPa or more in the bite [28] tends to force the lubricant out of the interface, so some asperity contact occurs simultaneously. Under mixed lubrication, both hydrodynamic effects and boundary friction/cohesion contribute to determining the overall friction coefficient [29]. The aim in lubricated rolling

is to push conditions as far toward the hydrodynamic side as practical – e.g. using higher rolling speeds or more viscous oils to minimize direct metal contact and friction [29-30]. However, there is a delicate balance: if friction becomes too low, the rolls may lose traction and the strip can slip (a condition sometimes called “no-grip rolling”). In practice, rolling lubricants are chosen and applied such that a stable mixed lubrication is maintained: friction is low enough to reduce force and wear, but high enough to maintain a forward tension on the strip and avoid excessive forward slip at the exit [30-31]. The concept of a neutral point in rolling (where strip speed equals roll surface speed) is also influenced by lubrication. Lower friction shifts the neutral point closer to the entry, resulting in less backward slip region and generally lower rolling force for a given reduction [32-33]. The Fig. 1.4 shown lubrication system in cold rolling process.

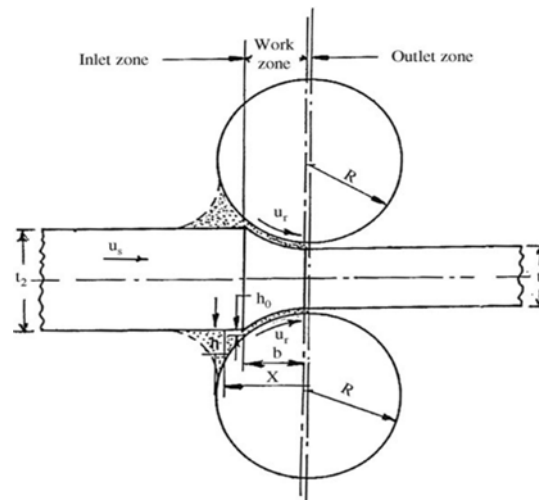


Fig. 1.4 Schematic diagram of cold strip rolling being lubricated with emulsion [30]

Where t_1 = outlet strip thickness; t_2 = inlet strip thickness; u_s =strip velocity; u_r =roll velocity

h_0 = minimum film thickness; h = film thickness; b = length of contact zone

X = coordinate along rolling direction; R = roll radius

1.3.2 Friction Constraints Governing Rolling Performance

One method utilised in the industrial sector to regulate foil gauge highlights the crucial role of lubricants in the rolling process. Due to the elastic deformation of the rolls, increasing the rolling load during foil rolling has only a limited effect on the reduction in foil thickness [34]. On the other hand, increased rolling speed alters the friction conditions in the roll bite through hydrodynamic lubricant entrainment. The entrained lubricant alters the shear conditions at the interface, reduces direct metal-to-metal contact, and consequently modifies the overall friction behaviour during the rolling process. This results in a measurable change in foil thickness [35].

Furthermore, when different lubricant or coolant flow rates are applied across the width of the roll, non-uniform cooling occurs. This leads to developments of temperature gradients along the roll width. These temperature variations lead to uneven thermal expansion and contraction of the roll material, which can influence the roll profile and contact conditions within the roll bite. Consequently, such gradients may affect the distribution of pressure, frictional behavior, and ultimately the dimensional accuracy and flatness of the rolled product [5]. Through the utilisation of the consequent differences in friction conditions, it is possible to control the reduction in thickness as well as the variations in form that occur over the breadth of the foil. The development of automatic gauge control (AGC) systems, driven by the growing demand for precise control of both gauge and form in foil rolling, as well as the need for higher rolling rates and improved productivity, has led to their creation. In response to detected shape abnormalities across the strip width, these systems adjust coolant flow patterns [28].

The friction conditions in metal rolling are governed by three primary constraints, which collectively control the interaction between the roll and the workpiece, influencing material flow, deformation behavior, and the efficiency of the rolling process.

i) In foil rolling, a bright surface finish is typically required, with strict limitations on surface defects such as pinholes. When the strip and roll surfaces are completely separated by a continuous lubricant film, the strip surface tends to roughen during elongation, promotes to the development localized depression known hydrodynamic pits [26,27].

Only when there is close and consistence contact between the strip and roll surfaces, a bright surface can be achieved without pitting defects [33].

ii) At the same time, a sufficient quantity of lubricant must be applied to limit frictional forces and to minimize damage to both the strip and roll surfaces, thereby reducing wear and extending tool life [24].

iii) In addition, adequate friction must be maintained to prevent skidding or slipping between the roll and the strip, which is essential for stable material transport through the roll bite and consistent thickness reduction [33].

Where surface finish is a critical consideration, these requirements collectively responsible that rolling should occur with partial surface contact combined with hydrodynamic lubricant action, a condition commonly referred to as mixed lubrication [34-37]. Consequently, lubricant properties, surface finish, and comprehensive rolling models play a central role in determining frictional behavior and product quality.

1.3.3 Lubricant Properties

While a Newtonian viscosity adequately describes the shear behavior of rolling mineral oils at low pressures and strain rates, it becomes inadequate at higher pressures and strain rates. At high pressures, two methods primarily assess oil characteristics. Bair and Winer [38, 39] prominently employed the first approach, which involves performing traction experiments within a specifically designed pressure vessel. Another way to determine oil quality is to measure traction in the nip of a concentrated contact lubricated with the oil. Evans and Johnson [40, 41] utilized this latter method to conduct extensive disc machine measurements for different oils. Disc machines are not perfect, but they are the only ones that can measure shear rates at high levels, and they are also relatively easy to operate. The findings from these two measurement methods provide the basis for a constitutive model of mineral oil. Oil can exhibit viscous fluid behavior up to a particular shear stress, plastic solid behavior up to a critical shear stress, or elastic solid behavior at sufficiently high shear rates; at this point, non-Newtonian behavior becomes relevant.

1.3.4 Surface Finish

The topology of the strip or foil surface determines two competing impacts. i) Water dynamic pits cut across the rolling direction are demonstrated by Ratnagar et al. [43] and Reid and Schey [42] when the film thickness exceeds the initial roughness by a significant margin. In a tensile test, which produces roughening of a similar kind, the orientation of the grains causes them to deform in different ways along the straining direction. Thomson [44] demonstrates the relationship between the grain structure, the workpiece's deformation history, and hydrodynamic roughening in a thick-film rolling operation. In lubricated rolling, Wilson [45] explores an additional mechanism to amplify this roughening. He demonstrates that, most likely due to grain structure, discrepancies in strip yield stress can lead to unstable hydrodynamic pressure, which in turn encourages the formation of these pits. (ii) In cases where the film thickness is significantly thinner than the roughness, hard roll asperities will crush any soft asperities on the workpiece. Less crushing occurs when the underlying material is not undergoing bulk deformation [46], but more crushing occurs when the bulk material is undergoing deformation, thereby increasing contact areas. In the absence of hydrodynamic activity and with lubrication provided by boundary additives, the sole factor influencing friction behaviour will be the expansion of contact areas. Both Stephenson [47] and Wilson [45] have conducted rolling analyses incorporating this type of friction and have determined friction laws for different asperity geometries in the absence of hydrodynamic effects. A good surface finish is required in the majority of metal-rolling processes, including foil rolling; thus, it is crucial to prevent hydrodynamic roughening to a significant degree. Because the film thickness between the roll and the strip must be equal to or less than the surface roughness height, the asperities will crush and come into contact with one another.

The significant interaction between the roll and the strip results in the smooth finish of the roll being transferred onto the foil. Fig. 1.5a and b present electron micrographs of a plastic replica derived from a roll utilized in the aluminum foil rolling process, alongside the surface of a piece of aluminum foil produced through industrial methods. The asperities are orientated circumferentially around the roll, a result of the grinding process employed to achieve the roll's surface finish.

The foil surface exhibits minor hydrodynamic roughening; however, the roughness structure predominantly reflects the longitudinal ground pattern of the rolls. The degree of pitting observed will be influenced by the specific conditions under which the strip has been rolled. In scenarios where other constraints take precedence over achieving a high-quality surface finish, pitting may be more pronounced. In specific experiments, a more pronounced level of hydrodynamic pitting was observed with thicker films. Fig. 1.6a and 1.6b present electron micrographs depicting these surfaces. In the micrograph presented in Fig. 1.6c, individual slip bands are discernible in the rolled lead. The imprinting of the roll pattern onto the foil is utilised in foil rolling, where the roughness of the ground roll surfaces in the pass schedule is reduced, leading to a progressive reduction in the foil's roughness. The characteristics of the strip, especially its propensity to develop pinholes, will be influenced by these hydrodynamic pits; however, the creation of oil pressure and friction may remain essentially unchanged.

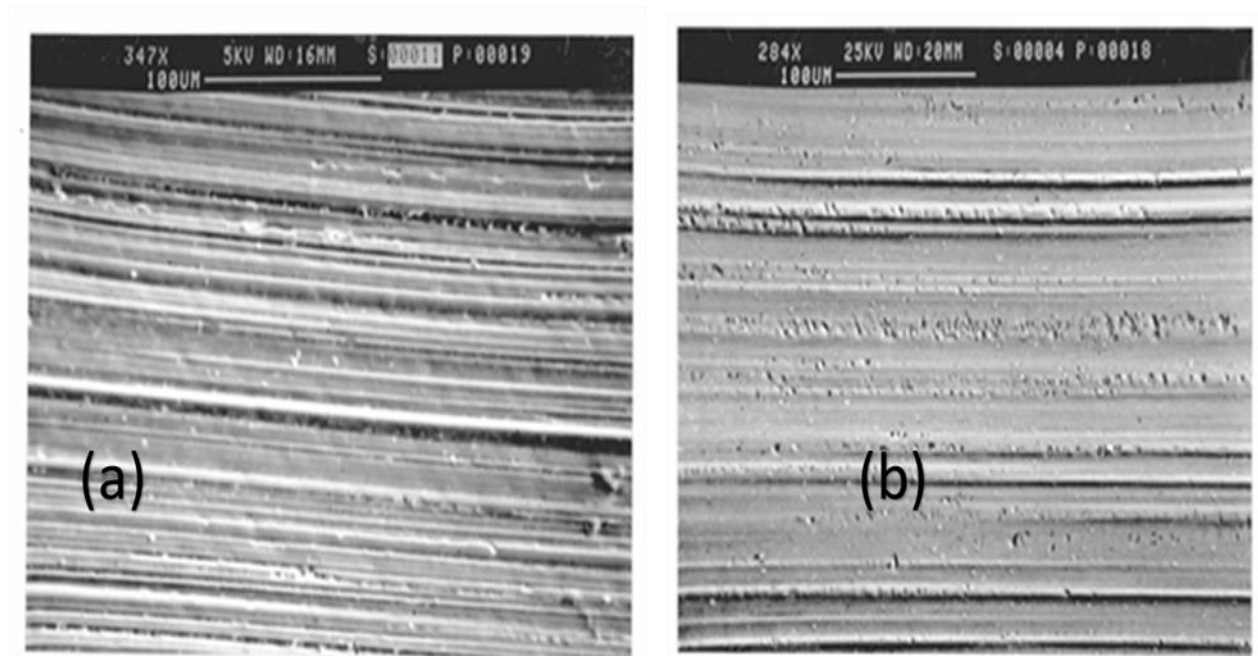


Fig. 1.5 Micrographs of a) Roll replica, b) Foil surface

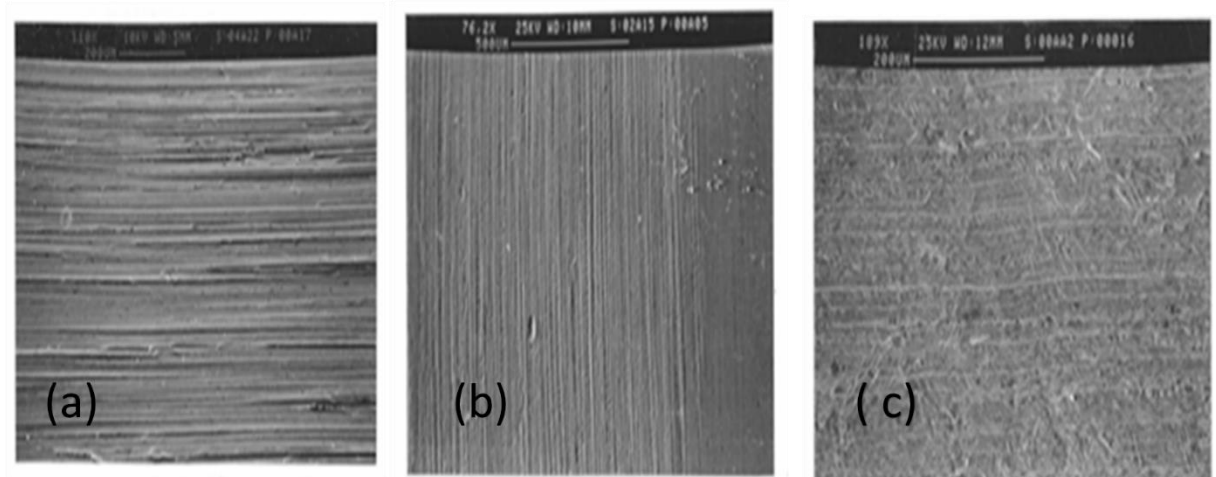


Fig.1.6 Micrographs of strip surfaces a) Undeformed strip b) Aluminium foil $A = 0.59$ c) Lead foil $A = 1.14$

1.4 Rolling Force Models

Rolling force models quantify the load required to deform the strip material plastically while accounting for elastic roll deformation, frictional resistance, and tension effects. Fundamentally, these models connect material properties, process geometry, and lubrication conditions into a predictive framework that can be validated against experimental rolling load measurements. The rolling force F is typically evaluated by integrating local contact pressures $p(x)$ along the contact arc between strip and work rolls. In analytical formulations such as those derived from Bland and Ford's 2D plane strain slab method, equilibrium equations in horizontal and vertical directions are applied to an infinitesimal element of length dx along the arc subtending angle $d\alpha$. The outcome reflects variations in flow stress due to progressive deformation rather than assuming a constant equivalent flow stress; this refinement better matches cold rolling where strain hardening develops throughout the bite. The local contact pressure distribution $p(x)$ is influenced significantly by friction behaviour and pre-tensions. Coulomb friction with coefficient μ modifies longitudinal stress gradients through a term proportional to $p(x)\mu$,

directly impacting the neutral point position where front slip changes to back slip along the arc. Friction coefficients are not constants but depend on lubrication regime, viscosity, sliding speed, and temperature, a coupling seen clearly when biodegradable lubricants with shear-thinning rheology shift Hersey number values even under nominally steady input parameters [169]. Variation of μ along the arc can be incorporated numerically into analytical models or resolved more directly in finite element simulations by linking surface tractions to a lubrication sub-model. Geometric inputs play a central role in determining rolling force through projected contact area and roll flattening effects. The Hertzian theory provides an initial estimate for purely elastic contact radius a between idealised roll and strip surfaces:

$$a = \sqrt[3]{\frac{3FR}{4E^*}}$$

where R is the radius of work roll and E^* is the composite Young's modulus defined as:

$$E^* = \frac{E}{2(1 - \nu^2)}$$

for identical roll and strip materials each with modulus E and Poisson's ratio ν . This leads to a nominal Hertzian contact pressure

$$P = \frac{F}{A} = \frac{F}{\pi a^2}$$

with A as projected elastic contact area. In practical cold rolling conditions, however, deformation within the strip introduces plasticity early along the arc; hence purely elastic Hertzian results underestimate actual contact lengths and force requirements. Consequently, semi-empirical adjustments or full slab analysis integration are preferred for accurate prediction. In slab analysis-based rolling force estimation, one evaluates:

$$F = b \int_0^{l_c} p(x) dx$$

where b is strip width and l_c is total projected contact length determined from reduction ratio, roll radius, and entry/exit angles. The variation of $p(x)$ incorporates strain hardening through $\sigma_f(\varepsilon) = K\varepsilon^n$, where K is strength coefficient and n work-hardening exponent. This function evolves from entry ($x = 0$) toward exit ($x = l_c$) as thickness reduces from t_0 to t_1 , meaning true strain varies as $\varepsilon_x = \ln(t_0/t(x))$. Field application demands measuring or estimating average $\bar{\sigma}_f$ value over the arc; older models used constants whereas refined approaches integrate continuously updated $\sigma_f(\varepsilon_x)$ values. Tension stresses at entry (σ_{x0}) and exit (σ_{x1}) directly alter net rolling load through superposition: entry tension effectively aids roll bite capture by reducing deformation demand at leading edge; exit tension resists elongation, lowering load near exit side but increasing it upstream depending on neutral point movement. Correct inclusion of these terms aligns predictions with experimental data. For SAE 1008 steel first-pass scenarios on GRL (green rolling lubricant), employing measured $\mu = 0.050$ with work roll diameter 460 mm yielded pressure maps consistent with analytical model expectations, highlighting that friction calibration improves predictive confidence across lubricant types [160]. Advanced computational implementations embed these force models within finite element packages where coupled thermo-mechanical solutions account for temperature-dependent viscosity influencing friction laws. This becomes essential for biodegradable emulsions whose viscosity drops markedly under asperity heating; under high reductions the resulting fall in $H = \eta U/P$, captured inside simulations as evolving boundary conditions at roll-strip interface nodes, shifts load distribution upstream, thus altering neutral point location in ways not anticipated by constant- μ , isothermal formulations. Waste particle intrusion modifies surface roughness mid-campaign, an indirect factor on rolling force since increased roughness amplifies ploughing component of friction. Bio-lubricants exhibiting strong polarity may adsorb onto fresh microcuts faster than mineral oils; while this helps restore film locally; it can slightly raise rolling load if adsorption coincides with debris accumulation zones acting as three-body abrasives [161]. Including such stochastic perturbations in computational force models remains challenging due to uncertain debris flux characterisation. Calibration of rolling force models against mill logs should

incorporate statistical estimation of uncertainty due to measurement noise in torque sensors, width gauges, tension meters, and lubricant flow monitors. Sensitivity analyses frequently reveal dominant influence from friction coefficient error bands; e.g., ± 0.005 change in μ can shift predicted force by several percent depending on reduction schedule, meaning tribute must be paid to accurate tribological characterisation in pre-run testing. Furthermore, field calibration may require periodic refresh due to seasonal viscosity shifts affecting real-life $\mu(H, T)$ relationships for bio-based stocks. In summary, rolling force models bridge material constitutive behaviour (strain hardening laws), process geometry (roll size, reduction), tribological state (friction law tied to Hersey number), and operational tensions into calculable projections of mill loading requirements. Foundational methods like Bland–Ford slab analysis enhanced by evolving flow stress description remain competitive for quick assessment; however, hybrid analytical–numerical treatments incorporating lubrication thermal-dependence are increasingly indispensable when unconventional lubricants such as biodegradable emulsions introduce non-linear rheological responses over the arc length [162].

1.4.1 Friction and Wear Models

Friction and wear models within the context of cold rolling provide a quantitative and mechanistic description of how interfacial forces, material removal, and surface degradation evolve over repeated roll-strip contact cycles. Building upon the load analysis, these models extend predictive capabilities by explicitly linking tribological regime boundaries, governed by parameters such as lubricant viscosity η , sliding speed U , and normal pressure P , to material loss rates, changes in surface morphology, and alterations in coefficient of friction (μ) over time. A common starting point for friction modelling is the decomposition of μ into contributions from adhesion between asperities (μ_a) and ploughing resistance around surface protuberances or embedded debris particles (μ_p). The total friction coefficient is expressed as:

$$\mu = \mu_a + \mu_p$$

where μ_a depends strongly on surface energy interactions (modulated by lubricant polarity and adsorption strength), and μ_p scales with the hardness differential between strip and debris, as well as with debris geometry relative to local contact pressures [163]. In biodegradable lubricants enriched with polar functional groups, stronger adsorption tends to reduce μ_a by promoting easier slip at metal–metal junctions within boundary lubrication (BL) zones. However, if filtration systems fail to remove fine abrasive contaminants efficiently, these same polar films may retain debris longer at the interface, elevating μ_p through increased ploughing interactions. Integrating equation into rolling simulations typically involves assigning spatially varying values along the contact arc to reflect transitions between BL, mixed lubrication (ML), and hydrodynamic lubrication (HL). The Hersey number $H = \eta U/P$ serves here as a regime classification variable; local decreases in H , whether from viscosity drop due to shear heating or rising P from reduction ratio increases, shift models toward higher μ values by expanding BL domain length mid-bite [164]. These shifts directly impact rolling power predictions and are critical for matching model outputs to measured torque data. Wear modelling in this context requires articulation of both adhesive wear processes, defined by junction growth and fracture mechanics under asperity loading, and abrasive wear from three-body particle action. A widely used relationship for adhesive wear volume V_a per unit sliding distance s is given by Archard’s law:

$$\frac{dV_a}{ds} = k_a \frac{F_N}{H_m}$$

where k_a is the dimensionless adhesive wear coefficient dependent on chemical compatibility between contacting surfaces, F_N is normal load, and H_m is microhardness of the softer material in contact. In cold rolling with steel work rolls against aluminium strip, H_m corresponds to aluminium’s near-surface hardness post-work-hardening; when biodegradable lubricants sustain ML/HL regimes effectively, asperity junction loading decreases and thus reduces V_a . Conversely, regime collapse expands adhesive junction

areas under high loads at asperities, raising values predicted by equation [166]. Abrasive wear volume per unit sliding distance can be similarly expressed:

$$\frac{dV_p}{ds} = k_p \frac{F_N}{H_m} \left(\frac{r_d}{r_c} \right)$$

where k_p is an abrasive wear constant linked to particle morphology, r_d is average debris radius, and r_c is critical radius above which particles indent beyond lubricant film thickness. For nanoparticle-reinforced bio-lubricants containing TiO_2 , ZnO , or h-BN inclusions at optimal concentrations ($\approx 0.2 - 0.3$ mass%), $r_d < r_c$, allowing particles to function beneficially as micro-roll elements rather than abrasives [167]. Agglomeration caused by poor formulation stability raises $r_d > r_c$, switching their role toward accelerated abrasion and increasing predicted V_p .

Temperature effects on both frictional components (μ_a, μ_p) and wear volumes are non-trivial for biodegradable lubricants with shear-thinning viscosity profiles. Localized heating within the roll bite can reduce lubricant viscosity, consequently decreasing film thickness to values below asperity heights in peak pressure zones at the mid-bite region. This facilitates an earlier transition to boundary lubrication (BL) conditions, even when the global feed temperature is maintained within nominal bounds ($25^\circ\text{C} \pm 2^\circ\text{C}$). [168]. Computational integration couples transient heat conduction in strip layers with dynamic viscosity-temperature behaviour drawn from rheometer characterisation; this enables prediction of where thermal excursions influence $\mu(H)$ enough to alter long-term wear rates beyond baseline expectations. Empirical calibration across industrial campaigns enforces realism within these models. Profilometry combined with scanning electron microscopy (SEM) validates predicted groove depths from abrasive term calculations against observed roll surface damage patterns after set tonnage throughput [169]. Adhesive wear estimations are enhanced through controlled pin-on-disc replication experiments employing representative alloy pairs under scaled loads (approximately 312 MPa contact pressure) and sliding velocities comparable to cold rolling conditions (0.5–1.2 m/s). These tests enable quantification of wear coefficients and junction growth rates

under boundary lubrication regimes. $\$$), both to determine constants k_a, k_p accurately for given lubricant formulations and to observe mode transitions when contaminants surpass designed filtration capacity thresholds [170]. An important stochastic component arises from irregular debris influx events caused by upstream process anomalies (e.g., strip edge cracking introducing hard oxide fragments). Including such discontinuous load multipliers into statistical wear models improves their predictive range substantially over purely deterministic forms; Monte Carlo simulation frameworks assign event probabilities based on historical campaign data while propagating resulting parameter perturbations through equations. This hybrid approach captures realistic variance bands observed in measured coefficients of friction throughout mill runs under biodegradable lubrication regimes [171]. Finally, integrating friction–wear sub models inside broader finite element analysis (FEA) packages allows simultaneous resolution of mechanical deformation fields with evolving interfacial state variables. Linking these sub models’ outputs, updated $\mu(x, t)$ distributions, to rolling force computations closes the loop between load estimation and damage prediction described earlier.

1.5 Background on Lubricants

Lubrication is among the earliest technological practices developed by humankind, with origins that predate most modern engineering systems. Archaeological findings indicate that lubricating substances were employed in ancient civilizations, particularly in Egypt, as early as the seventeenth century BC to facilitate the movement of heavy stone blocks during construction activities [48]. Historical records further suggest that animal fats such as tallow were applied to chariot axles around the fourteenth century BC, demonstrating an early understanding of friction reduction and mechanical efficiency. In large construction projects, including pyramid building, combinations of animal fats and water are reportedly used to assist material transport by reducing sliding resistance [49].

Before the advent of petroleum extraction, lubricants were predominantly **derived from natural sources**, including **vegetable oils and animal fats**, and they were extensively used

in early transportation and machinery systems. This reliance shifted significantly following the discovery of crude oil in Titusville, Pennsylvania, in 1859. This discovery marked the advent of large-scale petroleum production in the United States [48]. Petroleum-based lubricants rapidly replaced bio-based alternatives due to their lower cost, improved availability, and superior performance characteristics [50]. As industrial demand increased—particularly with the expansion of the automotive sector—lubricant formulations underwent systematic refinement. Solvent refining techniques emerged in the 1920s, followed by the widespread introduction of additives in the 1930s to enhance oxidation resistance, corrosion protection, and load-carrying capacity [52]. From the 1940s onward, additive technology became central to lubricant formulation, significantly extending service life and performance, especially in automotive applications. The development of synthetic lubricants in the 1950s further expanded lubrication technology, driven largely by the stringent requirements of aviation and aerospace industries [51].

Despite the dominance of petroleum-based lubricants, periods of global conflict and energy scarcity prompted renewed interest in vegetable oils as lubricants, fuels, and greases during the two World Wars. Later, the 1973 oil embargo highlighted the vulnerability of petroleum supply chains and intensified the search for alternative lubricants. In recent decades, escalating environmental concerns, stricter regulations, and volatility in fossil fuel prices have accelerated interest in cleaner and renewable lubrication solutions. During the 1980s, policy initiatives in Europe and growing industrial usage of biodegradable products in the United States during the 1990s further stimulated research into environmentally benign lubricants. Today, biodegradable lubricants are actively investigated for their renewable origin, low toxicity, and tribological performance comparable to, or in some cases exceeding, that of mineral oils [51-53]. Nevertheless, conventional vegetable oils exhibit limited thermal and oxidative stability at elevated operating temperatures, which restricts their direct application in demanding industrial environments [54]. To address these limitations, recent studies have explored the incorporation of nanoparticles into vegetable oils, demonstrating that optimized nano

lubricant formulations can significantly enhance friction and wear performance, approaching that of petroleum-based lubricants [53].

1.6. Vegetable Oils as Biodegradable Lubricants

Biodegradable lubricants have gained increasing prominence in recent years due to environmental regulations, sustainability objectives, and economic pressures associated with petroleum-based oils. Among various alternatives, vegetable oils have emerged as particularly attractive candidates owing to their renewable nature and favourable tribological properties. Commonly studied vegetable oils include sunflower, peanut, corn, coconut, soybean, canola, grapeseed, and jojoba oils, each offering distinct fatty acid compositions and lubrication characteristics. In general, vegetable oils exhibit low volatility, , elevated viscosity and high lubricity indices, making them suitable for a wide range of lubrication applications. Their biodegradability and non-toxic behaviour further enhance their appeal for environmentally sensitive industrial processes as Fig. 1.7.

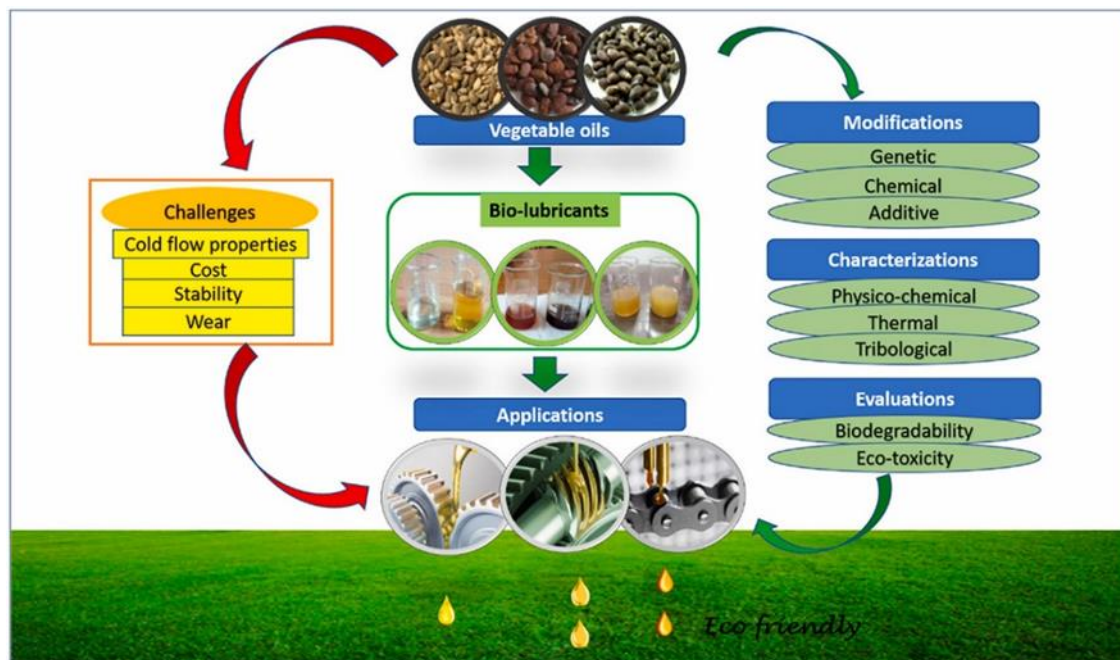


Fig. 1.7 Bio-lubricants from vegetable oils

Despite these advantages, the extensive industrial application of vegetable oils is constrained by several inherent drawbacks. One of the most notably issue is their poor

oxidative stability, which makes them prone to degradation and rancidity when exposed to heat, oxygen, or metal surfaces over time. And other issue is limited low-temperature performance. Oxidation in vegetable oils is primarily governed by free radical reactions, which can be mitigated to some extent by reducing free fatty acid content or modifying molecular structure. Additionally, vegetable oils typically possess a narrower viscosity range than mineral oils, restricting their operational flexibility under extreme conditions. Nonetheless, their renewability, reduced environmental impact, and decreased dependence on imported petroleum resources continue to drive research and development efforts.

From a tribological perspective, vegetable oils often outperform mineral oils in terms of lubricity due to the presence of natural ester functional groups, which promote strong adsorption onto metal surfaces [54]. They also exhibit significantly higher flash and fire points; for example, soybean oil has a flash point exceeding 600°F, compared with approximately 392°F for typical mineral oils. This characteristic enhances operational safety, particularly in high-temperature applications. Furthermore, vegetable oils contain fatty acids such as palmitic, oleic, and linoleic acids, which contribute positively to boundary lubrication behaviour by forming protective films at sliding interfaces [55]. In the earlier work, cottonseed, sunflower, neem oil, palm oil, coconut oil, peanut, and corn oils are examined as potential biodegradable lubricant alternatives, with particular attention to their fatty acid compositions [54] as summarised in Table 1.1.

Table 1.1 Fatty acid composition of vegetable oils [55]

Types	Fatty Acid						
Vegetable Oil	14:0	16:0	16:1	18:0	18:1	18:2	18:3
Cottonseed	1.1	27.3	1.4	3.1	16.7	50.4	-
Linseed	-	6.5	4.5	19.5	16.5	53.0	-
Palm	1.0	43.1	0.2	4.0	39.0	10.0	0.2
Sunflower	-	6.1	3.0	27.0	-	64.0	-

Soybean	-	12.0	3.6	23.7	51.4	8.8	-
Peanut	-	9.8	0.4	3.7	60.9	18.1	-
Corn	-	10.6	26.7	59.8	-	0.9	-

Table 1.2 Comprehensive Overview of Biodegradable Lubricants in Rolling Operations

Aspect	Description	Advantages/Limitations	Sources
Base Oils	Vegetable oils (soybean, sunflower, canola); synthetic polyol esters	High lubricity, viscosity index; poorer oxidative stability	[48-50]
Performance	Low friction/wear in cold/hot rolling; optimized via Taguchi-Grey methods	Comparable to mineral oils; industrial trials successful	[51-53]
Additives	Anti-wear (ZDDP), solid lubricants (boron nitride), nanoparticles (nano clay)	Improves wear resistance; enhances boundary lubrication	[54], [49-50]
Needs in Rolling	Reduce friction, prevent welding, control heat, surface protection	Replaces mineral oils; disposal/toxicity issues resolved	[55-56],[52]
Pros	Biodegradability, low toxicity, high flash point, cost savings on disposal	Environmentally superior	[48],[50],[52]
Cons	Oxidative instability, cold flow issues, higher cost	Requires antioxidants, pour-point depressants	[48], [50]

1.6.1 Sunflower Oil

Sunflower oil has recently attracted attention as a promising alternative to mineral oil-based lubricants due to its biodegradable nature and low environmental impact. Under moderate operating conditions, sunflower oil has been reported to exhibit satisfactory antifriction and anti-wear performance comparable to conventional petroleum-derived lubricants [57]. Its widespread availability, renewability, and eco-friendly characteristics further enhance its suitability for sustainable lubrication applications. Chemically, sunflower oil is composed predominantly of monounsaturated oleic acid (around 30%) and polyunsaturated linoleic acid (approximately 59%). The relatively high oleic acid content, compared with several other vegetable oils, contributes to improved oxidative resistance, which is advantageous for lubrication purposes [54]. In addition, the presence of palmitic acid in appreciable quantities has been shown to enhance friction-reducing behaviour under boundary lubrication conditions [55]. Sunflower oil also possesses a relatively high smoke point, typically close to 227 °C, indicating reasonable thermal tolerance during operation [59]. Nevertheless, to enable its broader adoption as a direct replacement for mineral oils in demanding applications, further enhancement of its oxidation stability through suitable additive technologies remains necessary.

1.6.2 Peanut Oil

Peanut oil is another vegetable oil of interest for lubrication applications owing to its favourable fatty acid composition and thermal characteristics. Compared with many other vegetable oils, peanut oil contains relatively high levels of oleic acid along with a significant proportion of palmitic acid, both of which are known to contribute positively to friction reduction and wear resistance [58]. Similar to sunflower oil, this oil exhibits high smoke point, typically around 230 °C, suggesting adequate thermal stability for moderate-temperature tribological applications [60]. In addition to its tribological potential, peanut oil is renewable, biodegradable, and widely available, making it an attractive candidate from both environmental and economic perspectives. However, like most vegetable oils, its susceptibility to oxidative degradation limits long-term performance, particularly at elevated temperatures. Consequently, further research is

required to improve its oxidation resistance through chemical modification or additive incorporation before it can be reliably employed as a substitute for conventional lubricants.

1.6.3 Corn Oil

Corn oil has also been explored as a biodegradable lubricant due to its advantageous fatty acid profile and sustainability attributes. It is characterized by relatively high concentrations of linoleic acid and palmitic acid, which play an important role in enhancing lubrication performance by promoting the formation of protective boundary films at sliding interfaces [58]. Corn oil exhibits a high smoke point, typically approaching 230 °C, indicating good resistance to thermal breakdown under normal operating conditions [60].

1.6.4 Neem oil

Neem (*Azadirachta indica*) oil serves as an effective biodegradable lubricant derived from the seeds of the neem tree, offering a sustainable alternative to petroleum-based oils in various industrial applications. Its natural composition, rich in fatty acids and antioxidants, provides excellent lubricity, reducing friction and wear by up to 70% in blends like those with castor oil, while exhibiting high oxidative stability and a favorable viscosity index. These properties make neem oil particularly suitable for machining, automotive, and maintenance uses, where it minimizes environmental impact through rapid biodegradation and outperforms mineral oils in cutting temperature reduction and surface finish quality.

Its renewable origin, environmental compatibility, and widespread availability further support its consideration as an alternative base stock for lubricants. Despite these advantages, the primary limitation of corn oil remains its inadequate oxidative stability, which can lead to rapid degradation under prolonged or high-temperature service conditions. Addressing this challenge through targeted additive development or



44 formulation strategies is therefore essential before corn oil can be effectively utilized as a replacement for mineral-based lubricants in industrial applications.

1.7 Background and Motivation

The study of cold rolling and tribological performance within mechanical engineering has increasingly shifted toward environmentally conscious approaches, driven partly by legislative pressure and partly by industrial competitiveness. Historical reliance on mineral oil-based lubricants (MB) has led to well-documented environmental challenges, including hazardous waste generation and atmospheric emissions due to combustion or disposal inefficiencies. These drawbacks stimulate the search for biodegradable cold rolling lubricants (BB) that can match or closely approximate the functional performance of MB while reducing ecological harm [62]. This shift is not trivial, as lubricant properties in rolling highly influence parameters such as force reduction, thermal management, and surface finish quality. From an environmental standpoint, long-standing concerns have been rooted in the pollutant load generated by petroleum-derived products in industrial processes. Studies comparing renewable-source lubricants with traditional mineral-based systems reveal marked reductions in smoke emissions during operational tests. For example, castor oil-based formulations evaluated under engine conditions produced smoke levels well below half that of mineral oils at identical load and velocity settings, meeting specifications. While those findings stem from engine operation rather than cold rolling per se, they illustrate transferable principles related to low volatility, cleaner combustion profiles, and reduced particulate output, characteristics highly relevant to rolling mill operational safety and compliance. From a production efficiency perspective, lubricant choice impacts work rolls forces directly. Comparative analyses between green rolling lubricants (GRL) and standard synthetic lubricants (SSL) have recorded average reductions in rolling force around 5.6 "%" when GRL are deployed [2]. Although variations exist due to equipment specifics, such as work roll diameter, these variations appear minimal compared with the lubricant's effect itself. Such reductions imply lowered energy consumption per ton processed, potentially influencing both economic metrics and

greenhouse gas inventory figures for the plant. In fact, large datasets suggest this force reduction trend holds statistically across multiple coil batches without substantial deviation due to geometric considerations [21].

CHAPTER 2

LITERATURE REVIEW

This chapter presents a comprehensive review of the existing literature related to the rolling process, lubrication mechanisms, and rolling forces. It also discusses various mineral-based and biodegradable lubricants used in cold rolling operations. Previous research findings and developments in these areas are critically examined. Based on the reviewed literature, the existing research gaps are identified. Finally, the objectives of the present thesis are formulated to address these gaps.

2.1 Introduction of Literature Review

There is also large volume of work available on traditional metal working fluids, the major pollutant being mineral oil based lubricants known for their low biodegradable characteristics, and toxic products formed in use [60]; thus research has been carried out to develop sustainable alternatives to mineral oil based lubricant's, namely vegetable oil based lubricants which imparts renewability and inherent lubricating properties [61]; although, research have shown that chemical modification of vegetable oils results in improvement of oxidative stability of vegetable oils which is one of the limiting factors of vegetable oils for industrial use [62]; however tribological performance of chemically modified vegetable oils in high pressure metal forming applications remains unexplored. Cold rolling involves complex lubrication mechanism as it includes mixed regime (asperity contact & fluid film) lubricated interface where both asperity contact and fluid film coexist [63]; adhesion theory of friction states that lubricants function primarily by separating sliding surfaces (total lubrication) using thin films that moderate the metal-to-metal contact [64]; therefore mineral oils have been able to provide effective separation between the sliding surfaces; however environmental persistence of mineral oils has engendered studies will be on bio-based lubricants. For example [65], was able operate at a friction coefficient of 22% less friction for their oils than SAE 10w oil in Aluminium forging indicating possible use for vegetable oils in metal forming. Recent developments in opacities have been exploiting palm oil derivatives. Palm oil derivatives offer a number of favourable properties as a lubricant, including balanced fatty acid composition and superior thermal stability [66]. Efforts to identify mechanisms of this action can be traced [67], who observed that during loading triglyceride molecules in palm oil have a tendency to form adsorbed protective films under pressure, and Montagnolli et al [69], who identified interaction of ester groups with metal surfaces that has the potential to reduce friction. These mechanisms are most relevant in aluminium cold rolling, where high contact pressures can lead to excessive wear of tooling and components. The quantifiable environmental gains that can be achieved with biodegradable lubricants in aluminium cold metal rolling were estimated by using life cycle assessments (LCAS). Walkman [61] showed that rapeseed-oil-based hydraulic fluids had 40% less global warming potential

compared to mineral oils, but higher eutrophication impacts; based on LCA, Montagnoli et al. [69] developed kinetic models showing faster of biodegradation rates for vegetable oils, with automotive lubricants being more readily depleted than the murkier and bruiser mineral counterparts. While indicating ecological and environmental gains of using bio-based formulations over lead & tin mineral oil, the above studies did not incorporate performance metrics of lubricants under industrial conditions. Aluminium processing in particular, Antocelli et al. [70] formed a natural based emulsion and showed that rolling forces could be reduced by 5.6% in steel cold rolling. Aluminium, with its higher ductility and tendencies to adhere present specific challenges not addressed in aluminium studies [71]. Layers of aluminium oxide formed during metal rolling complicate lubricating them, as the oxide sleeve formed around the area gets thicker and hence can either enhance or hinder lubricant effectiveness, dependent upon its morphology [72]. Rio et al. [73] studied and developed formulations of biodegradable lubricants for aluminium with additives, achieving a 26% reduction in friction by incorporating graphene nanoplatelets into an ester-based lubricant. Huo et al. [74] investigated the reduction of the rolling forces of a lubricant containing nanoparticles of 0.25–0.5% by weight intended for micro forming applications. Both methods are promising. However, concerns remain regarding toxicity and cost scalability to industrial commodity scale. The economics of sustainable lubricants from the production side is a contentious issue. While discreating savings in labour costs as a result of waste treatment reduce costs over the long term. They do not resolve issues for the producer, or mill manager, and Walker et al. [61] clearly show that this is a cost in connection with treating spent mwfs no matter what kind of lubricant is used. These three parameters of cost-performance-environment are where the chink in knowledge resides. For cold rolling aluminium, the economics of the process are often tight, so this paper builds on our previous work by conducting a side-by-side test of the performance of palm oil-based lubricant and conventional mineral-oil-based lubricants under actual cold rolling operating conditions. Our designs differ in that so much work in the field is performed only utilising environmental information or tribological information that are not fitted together into a useful whole, it integrates both aspects providing data that the aluminium

manufacturer can use. It not only provides information about friction co-efficient, and surface roughness and quality, but it also quantifies biodegradability and Chemical Oxygen Demand: metrics often omitted from metal forming work. Using the data present, a manufacturer would then be able to weigh operational performance against sustainability and come up with a bottom-line drawn over a rounded square. It shows that an emulsifying lubricant of palm oil can be as or more effective in offering environmental advantages whilst providing tribological performance staggeringly close enough; our 87% biodegradability results from our BB formulation is lightyears from what Adhvaryu et al. state, for example, for their modified soybean oils [66]. The presence of no statistically significant impairment of mechanical properties addresses a troublesome concern to an aluminium personally, raised obliquely by Huang et al. [61]. Rather than theoretical fantasies, it goes on to cogently-illustrate performance-dependant interstitial conditions to use or not use based on actual condition of the rolling mill when the performance gap shrink from both BB and MB lubricants, suggesting conditional guidance for the implement of the lubricants in the mill. It fills some of the chinks between the laboratory and industrial, ours is one of the first Ugandan-made. It wet allegiance with faith to the work force of our laboratory factory and this work indeed pushes the frontier towards true Ugandan aluminium.

Sargent and Stawson [75] evaluated lubricants with different viscosity and acid concentration on a 4-high laboratory cold rolling mill, finding that higher viscosity and acid concentration produced greater reduction and lower frictional power, with Sommerfeld Number being a key lubrication parameter. Kondo [76] investigated rolling stability parameters in Aluminium cold rolling, showing that lower viscosity oil, higher front tension, fatty acid additives, and surface roughness of 0.35-0.50 μm HCLA improved stability. Seng & Lenard [77] examined friction coefficient variation during cold strip rolling of Aluminium alloys without lubricant, finding that friction stress to normal pressure ratio was not constant, with rolling speed being the major parameter affecting friction variation. Lu and Chuang [78] studied surface roughness effects, showing higher

transverse roughness improved lubrication while longitudinal roughness caused poorer lubrication, with higher viscosity lubricants improving performance. Lin et al. [79] evaluated four lubricants during medium carbon steel cold rolling, finding coefficient of friction decreased with increasing reduction (0.28 to 0.05) and rolling speed significantly reduced frictional forces. Dohda and Wang [80] found that contact pressure was controlled by lubrication on the traction roll side while friction stress was controlled by the friction roll side. Baltov and Nedav [81] presented a validated model for contact friction in dry cold rolling. During hot rolling of Aluminum Hum et al. [82] measured friction, finding COF decreased with speed but increased with reduction. In their study, Tabary et al. [83] found that the amount of friction was highly dependent on the strip reduction as well as the ratio of film thickness to combined roughness

Saxena et al. [84] developed a hydrodynamic lubrication model showing uniform interfacial stress and 25-40% roll torque reduction with lubrication. The authors [85] found friction coefficient remained constant at low velocities but decreased at higher velocities, especially for high viscosity lubricants. Jeswiet [86] studied friction in different directions during Aluminium rolling, finding friction existed in both transverse and rolling directions. Luo and Keife [87] developed a thermal model showing maximum lubricant temperature occurred at exit for low-medium speeds and at entry for high speeds. Colás et al. [88] examined hot rolling work roll damage, finding cracks from thermal/mechanical fatigue with oxidation leading to high wear rates. Plouraboue and Boehm [89] analyzed Al 5182 sheet roughness, finding anisotropic self-affine surfaces with decreased roughness transfer after rolling. Qui et al. [90] developed an analytical model using Reynolds equation and mixed lubrication theories. Shirizly and Lenard [91] compared various lubricants, finding loads were unaffected by lubricant choice. McConnell and Lenard [92] tested six oils, with synthetic hydrocarbon producing lowest forces and friction. Ray et al. [93] investigated premature roll failures, finding higher retained austenite and tensile residual stress caused spalling. Liu et al. [94] measured rolling friction using sensor-embedded rolls, showing friction was not constant in the roll bite.

Ahmed and Sutcliffe [95] studied surface pit evolution, confirming hydrostatic pressure's role in pit elimination.

Le and Sutcliffe [96] developed a model for Aluminium foil rolling showing asperity flattening and decreased load with increasing speed. Lee et al. [97] created an integrated thermo-mechanical model validated by S.H.P.B. testing. Pawelski [98] observed three frictions in homogeneities improving prediction accuracy. Wang et al. [99] used volatile lubricants showing decreased friction with velocity. Le and Sutcliffe [100] presented a two-dimensional mixed lubrication model with two-wavelength surface roughness. Richelsen and Tvergaard [101] performed 3D analysis studying width spread effects. Lenard [102] found higher roll roughness increased friction at high speeds due to starvation. Tieu and Liu [103] measured friction variation showing non-constant coefficient along contact length. Azevedo & Neto [104] investigated roll failures, finding inadequate hardening depth and spheroidised pearlite promoting cracks. Azushima et al. [105] developed a hot rolling simulation showing additive effects on friction. Jiang and Tieu [106] showed work roll edge contact significantly affected wear. Jiang et al. [107] modelled friction variation effects on strip shape. Singh et al. [108] analyzed minimum film thickness at various starvation levels.

Byon et al. [109] developed a model to determine friction coefficient and flow stress-strain curves. Liu et al. [110] found EDT surface had anisotropic frictional properties with highest friction when sliding paralleled rolling direction. Jiang et al. [111] studied oxide scale effects showing roughness increased with interface friction and temperature. Fengshan [112] introduced a model with friction element layer between roller and workpiece. Hubert et al. [113] showed edge behaviour depended on forward slip with strain hardening affecting edge cracking. Li et al. [114] found 4% Cr + Ti material more wear resistant than 4% Cr alone. Ding et al. [115] presented a model for predicting grain refinement validated experimentally. Xie et al. [116] studied micro rolling showing oil film thickness increased with roll velocity and foil roughness decreased with pass reduction. Nana et al. [117] determined optimal parameters for copper alloy rolling: oil viscosity 40-50 mm²/s, pass

reduction $\sim 30\%$, and initial oil concentration 0.02-0.05. Huang et al. [118] showed AA3104 microstructure transformation during cold rolling. Yakubu et al. [119] found neem seed oil performed better than coconut/diesel mixture, with optimal mixture at 70% diesel. Li et al. [120] proposed a model for friction estimation with results closer to actual than SIMS model. Strasser et al. [121] developed a model using Archard's equation to predict strip wear. Literature survey reveals the need for accurate thermal and tribological analysis of lubricated roll-strip conjunction to optimize friction at high loads and speeds while achieving good surface finish, with lubrication analysis providing valuable information on scuffing, oil consumption, and wear.

2.2 Research Gaps

After an extensive review of the literature, the following research gaps were identified.

1. Absence of integrated thermo–tribological rolling models
2. Insufficient understanding of friction inhomogeneity and surface integrity
3. Limited integration of lubrication chemistry with rolling tribology
4. Lack of experimental validation under industrial rolling conditions
5. Inadequate evaluation of sustainable and bio-based lubricants in rolling
6. Absence of unified performance–environment–cost assessment frameworks
7. Unresolved multi-objective trade-offs in rolling lubrication

2.3 Need for Research

Aluminium cold rolling is increasingly operated at high speeds and large reductions, where lubrication becomes a primary control lever for roll bite friction, rolling load, strip surface finish, and process stability. In industrial practice, oil-in-water (O/W) emulsions and low-viscosity rolling oils are widely used; however, aluminium is highly sensitive to lubrication mode because surface chemistry and oxide formation interact strongly with the roll–strip contact conditions. For example, an emulsion's water content and concentration can directly change film formation behaviour and surface quality in cold-

rolled Al 5052, and lubrication can transition from plate-out to starvation as operating conditions intensify—creating a risk of lubrication failure at high reduction/high speed regimes [123].

At the same time, the shift toward more sustainable rolling fluids is constrained by aluminium-specific surface integrity requirements. While solution-type water-based lubricants are attractive for biodegradability and worker safety, they can exhibit poor lubricity and corrosion resistance, and have been shown to induce surface oxidation-related defects (e.g., roughness, non-uniform thick oxide layers, and loss of metallic lustre) on cold-rolled Al plates compared with oil-based lubrication [124]. This establishes a clear need to develop biodegradable oils/emulsions that preserve aluminium surface brightness and cleanliness while providing stable friction control.

Bio-based lubricants (e.g., palm and sunflower oils) have demonstrated measurable influence on rolling force and thickness formation during cold rolling of aluminium alloys (including 6xxx and 3xxx series), indicating practical potential but also underscoring the need for formulation- and condition-specific optimisation [125]. Moreover, ongoing studies on aluminium continue to confirm that friction conditions significantly affect process outcomes, motivating tighter control and validation under realistic rolling conditions [125].

This motivates the present work, which aims to experimentally and analytically compare the lubricated cold rolling performance of mineral and bio-degradable oil-based emulsions and to establish guidelines for their sustainable implementation.

2.4 Research Objectives

1. Experimental study for comparison of lubricated cold rolling process efficiency using mineral and bio-degradable oil-based emulsions.
2. Evaluation of mechanical and tribological properties of finished strips using mineral and biodegradable oil-based emulsions.

CHAPTER 3

METHODOLOGY AND EXPERIMENTATIONS

This chapter presents the methodology and experimental details used to accomplish the objectives of the thesis. In this chapter, the first part describes the tribological tests performed on a tribometer using different lubricants. In the second part, the cold rolling process was performed on a laboratory cold rolling mill. In the third part, the mechanical properties of the rolled samples were evaluated.

3.1 The Tribological Behavior of Aluminum 6061 and 6063 and EN31 Steel

Selection and Preparation of Work-Piece Material

- Aluminium alloy (e.g., Al 6061/ 6063 strips) as the test material
- Measure initial mechanical properties (yield strength, UTS, hardness, elongation) and surface condition (Ra) of the strips before rolling.

Selection and Formulation of Lubricants and Emulsions

- One conventional mineral oil-based (MB) lubricant and one or more biodegradable oil-based (BB) lubricants/emulsions were used for comparison of tribological properties.
- Prepare oil–water emulsions at selected concentration levels (e.g., 2–6% by volume), ensuring controlled droplet size and stability via proper mixing and filtration.

Cold Rolling Test Setup and Process Parameters

- Conduct rolling experiments on a laboratory or pilot cold rolling mill, with controllable reduction ratio, rolling speed, and lubrication conditions.

Vary key parameters:

- Rolling speed (low, medium, high range representative of industrial practice).
- Reduction ratio (e.g., 10–40% thickness reduction).
- Emulsion concentration and inlet temperature.

The tribological behavior of aluminum 6061 and EN31 steel was examined in situ under both dry and starved lubricated conditions. Frictional and wear properties play a crucial role in everyday life of human beings. It influences both energy consumption and the service life of products. To study these properties a Pin-on-disc tribometer was used to analyze the coefficient of friction (COF) and the specific wear rate (SWR) of the in-situ material having relative motion. The material of the disc was steel EN31, and the material of the pins were Al 6061 and Al 6063. Negligible wear of EN31 steel disc was assumed in comparison to the pin of both Aluminium. The material composition was as per Tables 3.1, 3.2, and 3.3.

Table 3.1 Composition of Aluminum 6061 Pin

Component	Al	Mg	Si	Fe	Mn	Ti	Zn	Ni	Cr	V
% value	98.402	0.6184	0.5787	0.2879	0.0077	0.0072	0.0526	0.0255	0.0103	0.0006

Table 3.2 Composition of Aluminum 6063 Pin

Component	Mg	Cr	Mn	Zn	Si	Cu	Fe	Ti	Sn
% Value	0.5602	0.0584	0.0834	0.0244	0.5384	<0.015	0.3111	0.0108	0.0441
Component	Pb	Ni	Be	Sr	Zr	Ca	V	Al	
% Value	0.0324	<0.020	<0.0001	<0.0005	<0.0030	<0.001	0.0112	98.3153	

Table 3.3 Composition of steel EN 31 Disc

Component	Fe	Cr	Mn	C	Si	Mo	Ni	Cu	Al
% value	96.8898	1.0942	0.6514	0.4207	0.2828	0.2449	0.1193	0.0681	0.0513
Component	V	P	S	As	Co	Pb	B	W	
% value	0.0469	0.0339	0.0286	0.0259	0.0226	0.0141	0.0127	0.0018	

The micro hardness of the specimens was assessed using a Vickers micro hardness tester (Fig. 3.1). The specimen was carefully polished to ensure a smooth surface and then securely positioned on the platform of the testing apparatus. A specific load of 0.30 N (equivalent to 0.03 kg) was applied for 120 seconds. The specimen surface exhibited an impression created by the Vickers hardness indenter. Hardness values were measured at



various positions on the specimen surface. The arithmetic mean of all the measured values was then calculated to obtain a representative average. The evaluation was conducted on the EN31 steel disc and Aluminum pin specimen. The micro hardness measured for the Aluminum pin was 33.0 HV, while the EN31 steel disc exhibited a micro hardness of 2004.9 HV.



Fig. 3.1 Measurement of Vicker Hardness



3.2 Specifications of EN31 Steel Disc and Aluminum Pin Samples

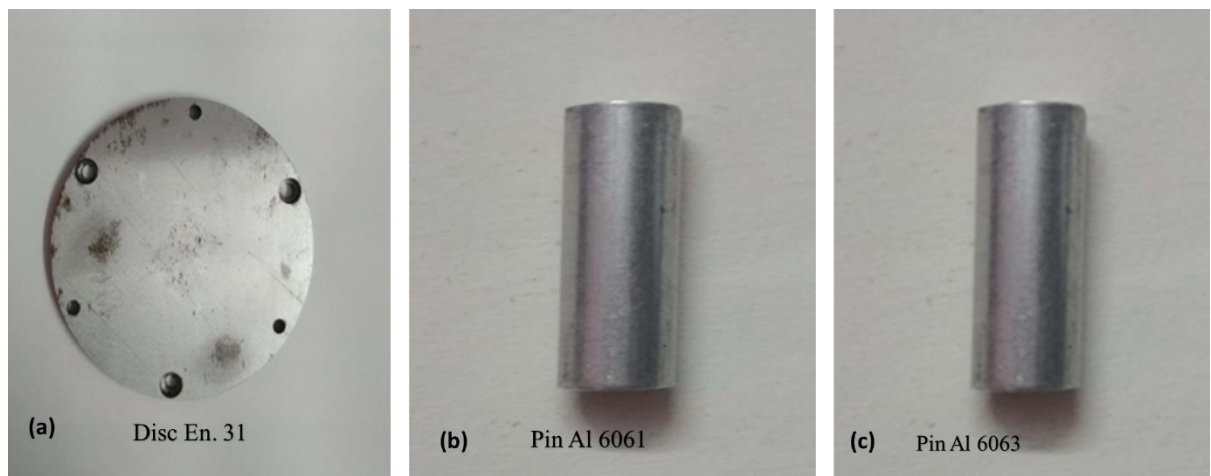


Fig. 3.2 (a) Bottom Specimen, EN31 Disc 100 mm diameter. (b) Top Specimen,

Aluminum 6061. (c) 6063 similar cylindrical pin 8mm diameter and 30mm length

A disc made of EN 31 steel was selected for experimentation, as illustrated in Fig. 3.2 (a). The disc surface was initially prepared by surface grinding, followed by lapping. The surface roughness was evaluated using a stylus surface roughness tester at radii of 20, 30, 40, 50, 60, and 70 mm in the circumferential direction, as well as at 60 degrees in the radial directions from a reference line (Fig. 3.3). The measurements were conducted on five surfaces of the disc and five samples of the aluminium pin. The measured average surface roughness Ra values were 0.51 and 0.44 μm , respectively.

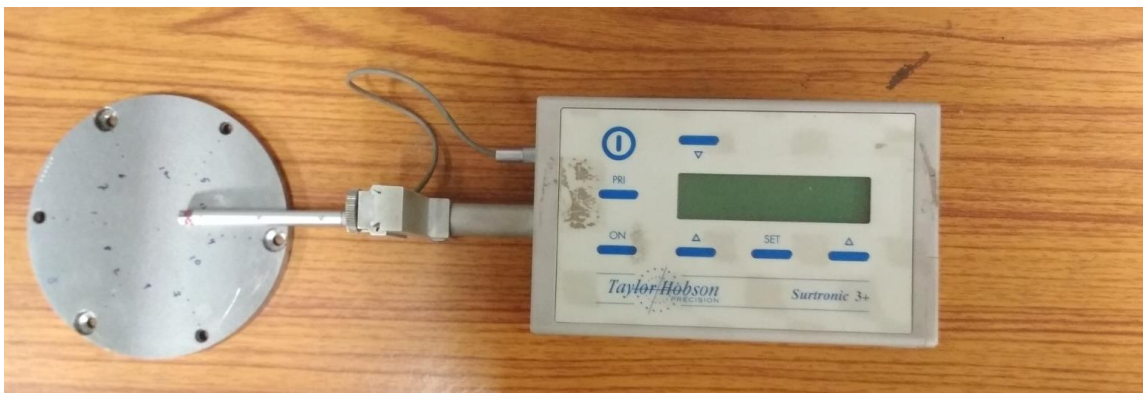


Fig. 3.3 Surface roughness checking on stylus Surface Roughness Tester

A cylindrical Al 6061 pin with a diameter of 8 mm and a length of 30 mm was utilised for testing on an EN31 steel circular disc, as illustrated in Figure 3.2(b) (c). The cylindrical pin face was meticulously prepared by rubbing the pins with fine-grade emery paper, followed by careful removal of debris with soft tissue. Flatness was assessed using an ink-impression method. To reduce bending, the aluminium cylindrical pin was securely clamped, leaving only 4mm exposed for the test on the EN31 steel disc. An aluminium rolling lubricant was utilised for starved lubrication between mating pairs. The characteristics of the lubricant employed are detailed in Table 3.4.

Table 3.4 Properties of Rolling Lubricant

Property	Value
Kinematic Viscosity at 40°C, (c St)	2.4
Flash Point, °C, (COC), (Min)	80
TAN, mg KOH /gm	0.002

Distillation Range,	
a) IBP	205
b) FBP	242

3.3 Description of Pin on Disc Test Rig

A high-temperature rotary tribometer was utilized for the testing rig in accordance with ASTM G-99 standards. The lower specimen is secured to a circular disc that is driven by a motor operating at different speeds. The speed varies from 300 to 3000 rpm. The specimen is secured to the circular disc by four bolts. The upper specimen of Pin has been secured in the specimen holder in a vertical orientation. Weights were placed on pans with a horizontal platform aligned with the direction of gravity for loading. The forces were conveyed via a lever mechanism. The details of the wear test rig are presented in Table 3.5.

Table 3.5 Specification of the Pin on the Disc Tribometer

Parameter	Value
Pin size (mm)	Diameter of 3,4,6,8,10 &12
Disc size (mm)	Diameter ϕ 100 x 8 and ϕ 165 x thickness 8
Wear track diameter (mm)	Min 20 and max 80 in steps of 2
Disc speed (rpm)	Min 300, max 3000
Normal load (N)	Min 0.1; max 200; least count 0.1
Wear (μ m)	0-2000 in the steps of 1, LVDT
Disc speed sensor (rpm)	300-3000, proximity type
Normal load range (N) and sensor	20-200 N, Dead Weight
Frictional force (N)	0-200, Load Cell

3.4 Experimental Process

The high-temperature rotating tribometer underwent wear tests in accordance with ASTM G99, as illustrated in Fig. 3.5. The testing used five sets of EN31 discs and 50 samples of pins. The test was first conducted in dry conditions, then in lubricant-free conditions. Tests were done on sliding speeds of 1.25, 1.5, 1.75, 2, and 2.5 m/s with a load of 20 N. The sliding distance was kept at 1500 m. The same steps were followed for loads of 25, 30, electronic balance, which had a minimum count of 0.001 g, was used to record the initial weight and the weight of the Aluminum pin after the test (Fig. 3.4). The same tests were

finished with starved lubrication conditions.



Fig. 3.4 Measurement of sample weight using an electronic weighing machine

The sensors sent the computer information about friction and wear. We used the weighing balance method to figure out the cylindrical pin's unique wear rate.

$$\text{Specific wear rate} = \frac{\text{Change in volume (m}^3\text{)}}{\text{Load (N)} \times \text{Sliding Distance (m)}} \quad (1)$$

$$\text{Change in volume} = \frac{\text{Weight loss}}{\text{Density of material}} \quad (2)$$

3.5 Design of Experiment

3.5.1 Taguchi method for process parameter optimization

The Taguchi method optimizes the data using engineering and statics techniques. The Taguchi optimization approach uses three conditions: "Larger is better," "Nominal is better," and "Smaller is better." An analysis and optimization of the required data is done by computing a signal-to-noise ratio (S/N). In this context "Signal" refers to the desired value (mean). "Noise" is the value that is unwanted (standard deviation). Thus, the ratio of the output parameter's mean to its standard deviation is known as the S/N ratio [127, 128]. The optimization condition is determined by the type of output parameters. Since the output parameters COF and SWR between tribo-pairs should be at their lowest, the smaller the better condition (equation 3) was used in this investigation.

Smaller the better

$$n = -10 \log_{10} \left(\frac{1}{n} \sum_{i=1}^n x_i^2 \right) \quad (3)$$

Where x_i is COF and SWR value for the i^{th} test in that trial.

3.5.2 Taguchi's design of experiment

The L_{16} orthogonal was developed using Taguchi's orthogonal array approach. The COF and SWR between tribo-pairs were determined for the optimum value of sliding speed, load, and fully flooded lubricating conditions. Four distinct process parameter levels were taken into consideration when designing the L_{16} orthogonal array. There were two output parameters: COF and SWR. Process parameters were sliding speed (s), load (W), and lubricant type (LT) (Table 3.6, 3.7).

Table 3.6 Process parameters and levels applied in the DOE

Process Parameter	Symbols	Units	Levels			
			1	2	3	4
Lubricant Type	LT	-	Fully flooded (FF) Neem Oil	Fully flooded (FF) Rolling Lubricant	Fully flooded (FF) Coconut Oil	Dry
Sliding speed	s	m/s	1.25	1.5	1.75	2.0
Load	W	Kg	4	6	8	10

Table 3.7 L_{16} Design matrix and results

Test No	Process Parameters				Output Parameter			
	Lubricant (LT)	Type	Sliding speed (s) m/s	Load (W) Kg	COF	SWR $\text{m}^3/\text{Nm} \times 10^{-9}$	S/N (COF)	S/N (SWR)
1	Neem Oil (FF)		1.25	4	0.3227	0.1557	9.8240	16.1560
2	Neem Oil (FF)		1.5	6	0.5012	0.7170	5.9998	2.8899
3	Neem Oil (FF)		1.75	8	0.6202	1.3041	4.1494	-2.3064
4	Neem Oil (FF)		2	10	0.4842	1.6003	6.2995	-4.0839
5	Rolling Lubricant (FF)		1.25	6	0.0874	0.0504	21.1698	25.9545
6	Rolling Lubricant (FF)		1.5	4	0.0967	0.0016	20.2915	55.8375
7	Rolling Lubricant (FF)		1.75	10	0.0887	0.2010	21.0449	13.9342
8	Rolling Lubricant		2	8	0.0854	0.0181	21.3708	34.8532

	(FF)						
9	Coconut Oil (FF)	1.25	8	0.0165	0.0614	35.6767	24.2419
10	Coconut Oil (FF)	1.5	10	0.0097	0.1058	40.2646	19.5127
11	Coconut Oil (FF)	1.75	4	0.0561	0.0123	25.0156	38.2213
12	Coconut Oil (FF)	2	6	0.0092	0.0136	40.7242	37.3520
13	Dry	1.25	10	0.0080	0.0145	41.9382	36.7527
14	Dry	1.5	8	0.0045	0.0142	47.0328	36.9479
15	Dry	1.75	6	0.0090	0.0078	40.9635	42.2127
16	Dry	2	4	0.0103	0.0032	39.7433	49.8169

3.6. Description of Tribometer

A standard pin-on-disc tribometer (Fig. 3.5) was used, conforming to ASTM G-99 specifications. The apparatus includes:

- Rotating Disc Holder: The EN31 steel disc was clamped horizontally and driven by a variable-speed motor (range 200–2000 rpm).
- Pin Holder: The Al 6063 pin was held in a vertically oriented holder, allowing for free sliding contact at the lower face.
- Loading Mechanism: A lever arm and pulley system permit dead weights of 4, 6, 8, or 10 kg to be applied as normal load on the pin.
- Frictional Force Sensor: A beam type load cell arrangement was employed to measure the frictional force between interface.
- Wear Measurement: A linear variable displacement transducer (LVDT) was used for wear measurement. In this study, it used the weight-loss method for wear calculation. [127].

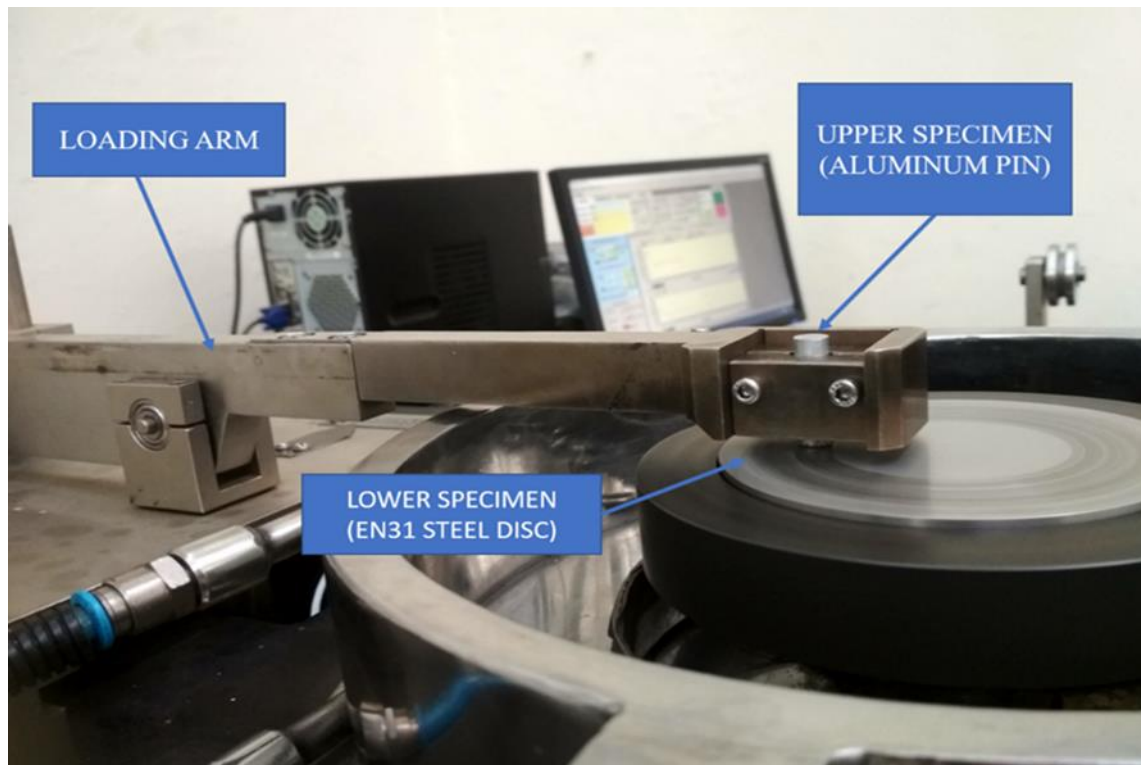


Fig. 3.5 Set up of Tribometer

The experimental methodology was meticulously crafted to systematically assess the performance of biodegradable palm oil-based emulsions (BB) in comparison to conventional mineral oil-based emulsions (MB) within the context of aluminum cold rolling. This section delineates the technical specifics of material selection, experimental setup, and characterization techniques.

3.7 Material Selection

3.7.1 Aluminum alloy selection

Commercial purity aluminium alloy AA1100 was used for the workpiece material due to its prevalent use in packaging and industrial applications. The initial strip dimensions were standardised at 200 mm × 50 mm × 3.0 mm according to ASTM B209

specifications. To eliminate any contamination, all strips were degreased in acetone prior to rolling and dried for good lubrication.

3.7.2 Lubricant selection

Two lubricant systems have been investigated:

- **Mineral Oil-Based Emulsion (MB):** A commercially available cold rolling oil-in-water emulsion with 2% oil concentration, comprising paraffinic base oil and proprietary friction modifiers.
- **Biodegradable Oil-Based Emulsion (BB):** A custom-formulated palm oil-based emulsion (2% oil concentration) using refined, bleached, and deodorized (RBD) palm oil stabilized with non-ionic emulsifiers.

The viscosity of both emulsions was measured at 40 °C using a rotational viscometer, while biodegradability was assessed in accordance with the OECD 301F standard for evaluating the ready biodegradability of lubricants and surfactant-based formulations [129].

The present invention concerns an oil-in-water lubricant emulsion intended for the cold rolling of light metals, with particular emphasis on aluminum materials, encompassing both commercially pure aluminum and aluminum-based alloys [130-132]. In comparison with conventional oil-based rolling lubricants, oil-in-water emulsions provide significantly enhanced cooling performance due to the high latent heat of vaporization of water. This improved heat dissipation capability enables higher thickness reductions per rolling pass and/or allows operation at increased rolling speeds without compromising strip quality [133].

Beyond these economic advantages, which directly contribute to improved process efficiency and industrial rationalization, aqueous lubrication systems also offer important environmental benefits. These include a substantial reduction in waste gas emissions and a decreased dependence on mineral oil resources, aligning with increasingly stringent environmental regulations imposed on metalworking fluids [133]. As a result, oil-in-water emulsions have attracted considerable attention within the light-metal processing sector. In particular, the aluminum industry has undertaken extensive experimental and

industrial-scale investigations to evaluate the feasibility and performance of aqueous emulsion lubrication in the cold rolling of aluminum strips [134-135].

The Mineral Oil-Based Emulsion (MB) employed in this study is a commercially available oil-in-water (O/W) cold rolling lubricant, formulated at an oil concentration of 2 % (v/v). The emulsion comprises a paraffinic mineral base oil, proprietary friction-modifying additives, emulsifiers, and corrosion inhibitors, specifically designed to ensure emulsion stability and effective lubrication under the high contact pressures and severe shear conditions encountered during cold rolling operations [136].

i. Water Phase Preparation

Clean, deionized or filtered industrial water is used as the continuous phase to minimize contamination and enhance emulsion stability. The water temperature is typically maintained at ambient conditions (25–30 °C) to facilitate effective mixing.

ii. Oil Phase Composition

The oil phase comprises:

- Paraffinic mineral base oil providing load-carrying capacity and boundary lubrication.
- Proprietary friction modifiers (e.g., fatty acid derivatives, polar additives) to reduce roll–strip friction.
- Emulsifying agents (non-ionic or anionic surfactants) that lower interfacial tension between oil and water.
- Minor additives such as rust inhibitors and anti-oxidants.

iii. Emulsification Process

The emulsion is prepared by slowly adding the mineral oil concentrate into water under continuous agitation. A mechanical stirrer or high-shear mixer is employed to break the oil phase into micron-sized droplets. High shear forces promote:

- Uniform dispersion of oil droplets
- Activation of surfactant molecules at the oil–water interface
- Formation of a stable oil-in-water emulsion

The oil-to-water ratio is carefully controlled to achieve a final oil concentration of 2%, ensuring reproducibility and consistent lubrication performance. The flow process diagram for preparing of Emulsion is shown in Fig. 3.6 below:

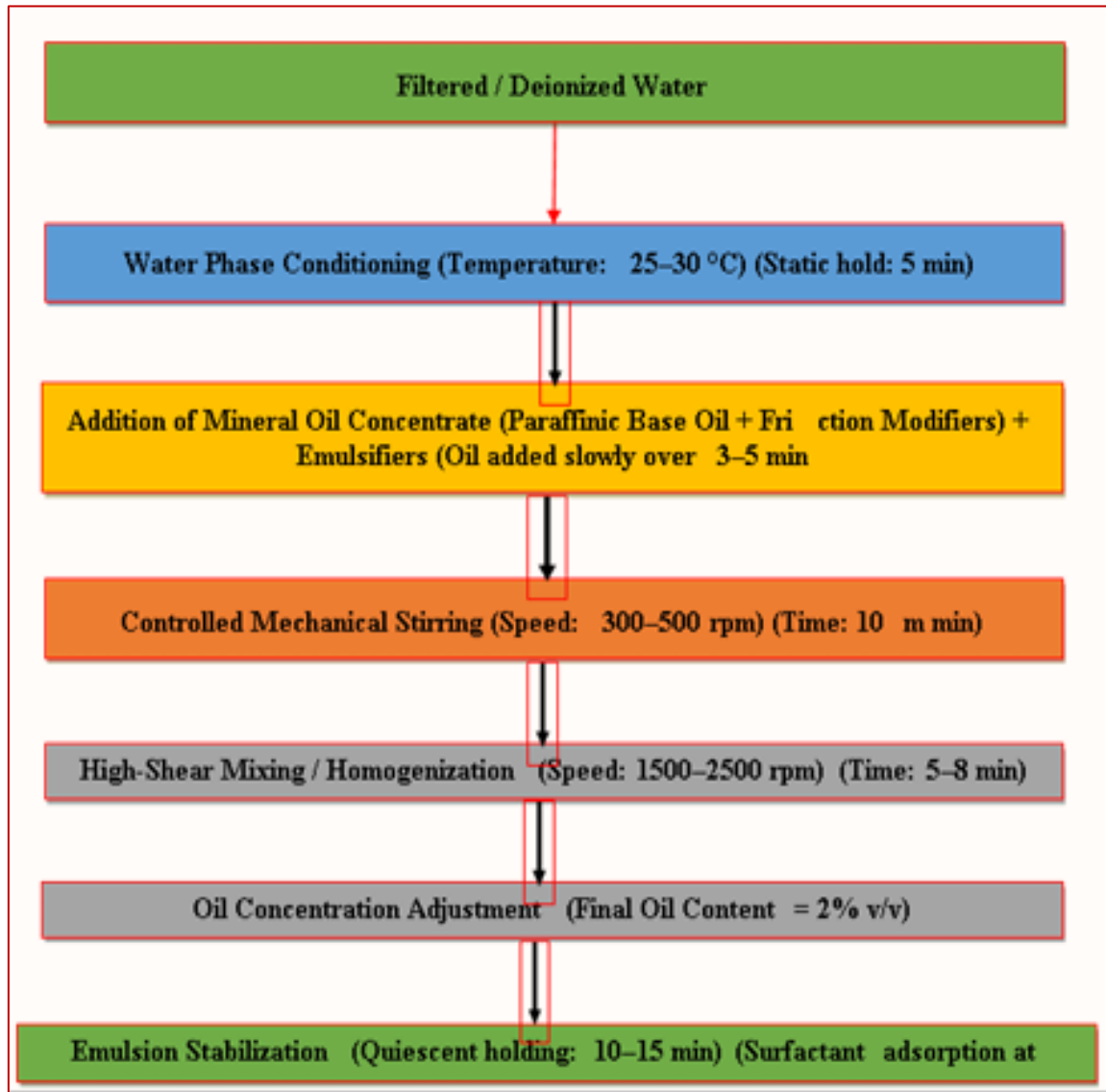


Fig. 3.6 Flow process diagram for the preparation of an Emulsion

iv. Stabilization and Conditioning

After mixing, the emulsion is allotted to stabilize for a short duration to ensure complete surfactant adsorption and droplet size uniformity. The resulting emulsion exhibits:

- Good kinetic stability against creaming or phase separation
- Low effective viscosity, aiding heat dissipation
- Adequate lubricity, enabling mixed and boundary lubrication regimes during cold rolling

v. Functional Role in Cold Rolling

During rolling, the water phase primarily acts as a coolant, while the dispersed oil droplets are entrained into the roll–strip interface. Under high contact pressure, oil droplets deform and coalesce locally, forming a thin lubricating film that:

- Reduces friction and rolling load
- Minimizes roll wear
- Enhances the surface finish of the rolled strip.

3.7.3 Cold Rolling Mill Configuration

In this subsection, the description of a lab-scale cold rolling mill is reported, in which the two rolls (150 mm diameter × 60 mm barrel length) were made of AISI D2 tool steel, hardened to 60 HRC for wear resistance. The rolling loads are monitored using load cells ($\pm 0.5\%$ accuracy); additionally, torque sensors are used to calculate power consumption, while LVDTs are used to track thickness reduction. The raw data is sampled at 1000 Hz by a high-speed data acquisition system.

3.7.4 Lubrication Delivery System

A closed-loop emulsion delivery system was developed, consisting of a 20-liter reservoir tank, centrifugal pump, and precision spray nozzles. The emulsion flow rate was maintained at 2 liters per minute, with temperature controlled at $25 \pm 2^\circ\text{C}$ using a PID-regulated heat exchanger.

3.7.5 Tribological Characterization

The friction and wear behavior of the material were systematically investigated using a pin-on-disc tribometer (DUCOM TR-20LE) operating under normal loads of 10–40 N,

sliding velocities in the range 0.1–0.3 m/s and a sliding distance of 500 m. The friction coefficient μ was calculated from the tangential force/normal load ratio. Post-test surface roughness measurements were then performed with an accuracy of 0.01 μm using a stylus profilometer (Mitutoyo SJ-410. These measurements determined both the arithmetic mean roughness (Ra) and the peak-to-valley height (Rz) of the tested surface

3.7.6 Mechanical Property Evaluation

i. Tensile Testing: The tensile strength of the prepared samples was evaluated using a universal testing machine (UTM, Model: Tinius Olsen) with a loading capacity of 50 kN and a speed range of 0.001 mm/min to 500 mm/min. It is illustrated in Fig. 3.7.

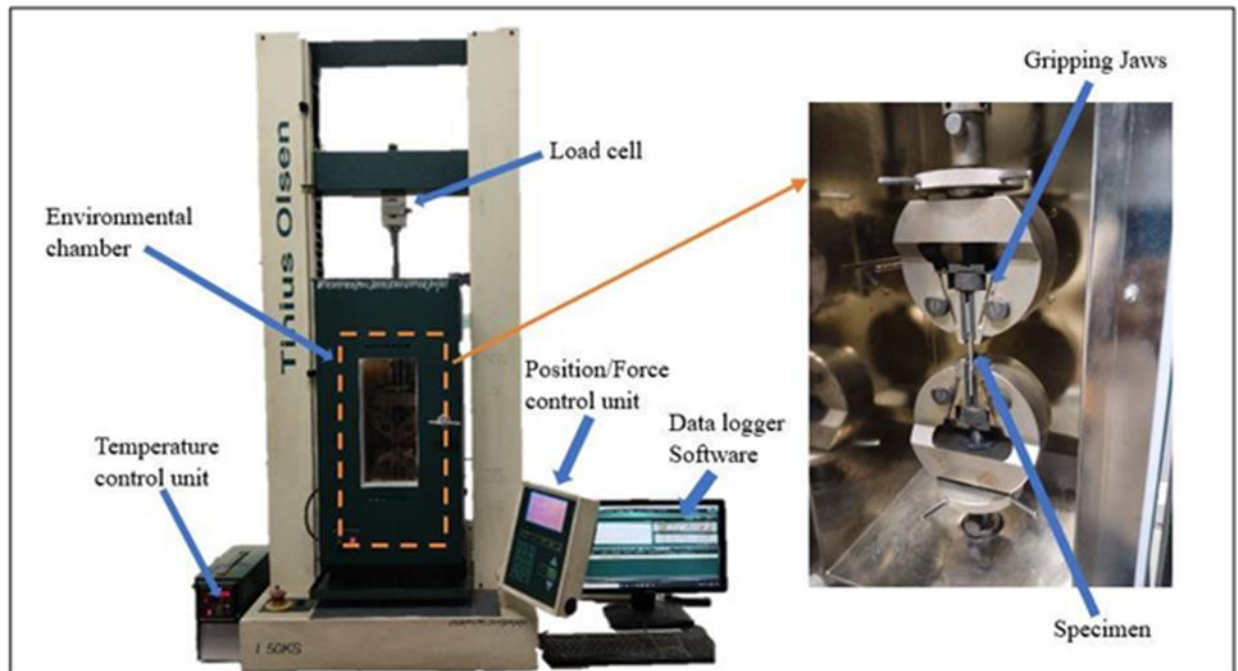


Fig. 3.7 UTM integrated with environmental chamber

The preparation stage includes careful deburring and removal of surface defects introduced during rolling to avoid stress concentration artifacts that could artificially reduce elongation measurements. During testing, a uniform tensile load is applied until fracture, with continuous measurement of load F_{load} and extension ΔL . True stress σ_{true} and true strain ϵ_{true} are derived via:

$$\sigma_{\text{true}} = \frac{F_{\text{load}}}{A_i} \quad ; \quad \epsilon_{\text{true}} = \ln \left(\frac{L_0 + \Delta L}{L_0} \right)$$

where A_i is instantaneous cross-sectional area and L_0 initial gauge length. Conversion from engineering to true measures allows better correlation with plastic flow models used in thermomechanical simulations for cold rolling. Yield strength determination employs the 0.2% offset method to identify the departure from elastic linearity on the stress-strain curve; this threshold directly relates to onset of dislocation motion within the strip's microstructure [139]. Cold rolling alters these properties through work hardening, grain refining in surface-adjacent layers combined with increased dislocation density throughout the thickness. Trials comparing mineral oil-based lubricants (MB) with biodegradable palm-oil-based emulsions (BB) under identical reduction ratios (10–40%) demonstrate close parity in σ_y and UTS values, with deviations of approximately 3% for yield strength at a 30% reduction [137].

ii) Microhardness: The Struers Duramin-40 M1 Vickers microhardness tester (Hardness scale: 0.2 Hv and dwell time: 10 seconds) depicted in Fig. 3.8 was used to measure the microhardness of the produced nanocomposite. The mechanical qualities are tested at four different temperatures, including room temperature. As illustrated in Fig. 3.9, each tensile test is repeated five times to ensure that the experimental data points are consistent. HORIZON software records force and position data to calculate mechanical properties.

Using the force-elongation data, we constructed real stress-true strain curves and $\ln(\text{actual stress}) - \ln(\text{true strain})$ curves.

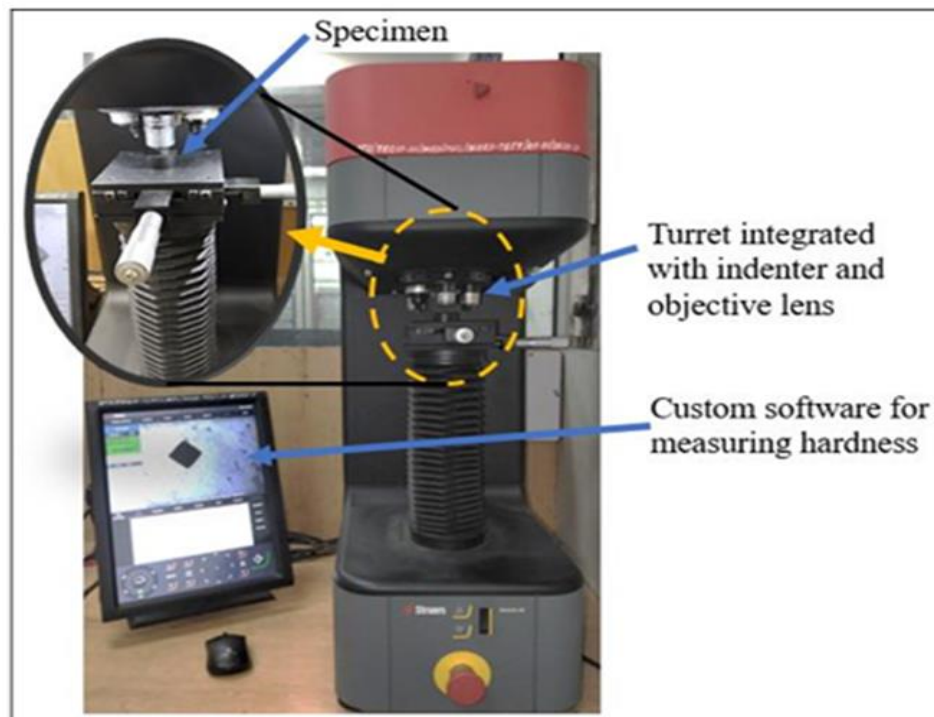


Fig. 3.8 Micro-Indentation Hardness tester

3.7.7 Experimental procedure

Cold rolling experiments were performed under the following parameters:

- Reduction ratios: 10%, 20%, 30%, 40%
- Rolling speeds: 5, 10, 15, 20 m/min.
- Lubrication conditions: dry, MB emulsion, BB emulsion

All experimental runs were repeated three times and the results were averaged. The parameters measured during rolling included rolling force and torque for all passes, and the exit thickness was measured for every pass. The product itself was characterised using surface roughness measurements, micro hardness mapping, and tensile testing to assess

the effect of lubrication. To determine environmental impact, the biodegradability (OECD 301F) and chemical oxygen demand (COD) according to ISO 6060. An economic analysis compared the cost of the lubricants using currently available prices for ingredients and processing for a sample. This comprehensive study enables a head-to-head comparison between conventional lubricants and biodegradable lubricants based on technical & environmental merits.

3.8 Tensile Test Specimen

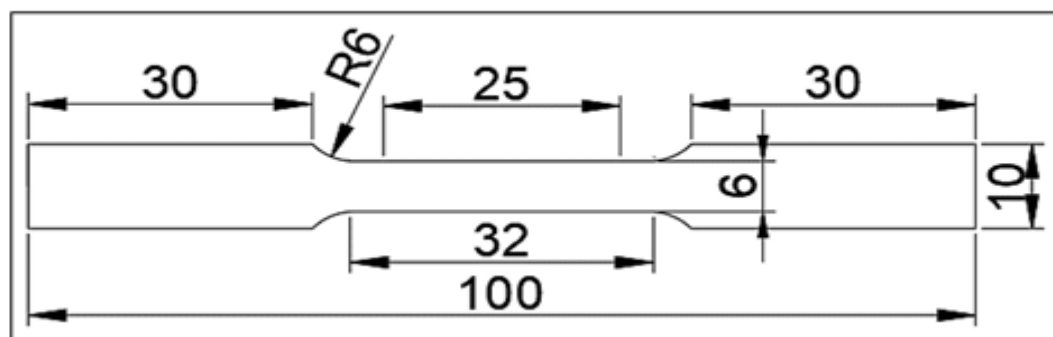


Fig. 3.9 Tensile test specimens (dimensions in mm)

Tensile test specimens were fabricated following with the ASTM E8/E8M-09 sub-size standard, with the tensile axis aligned parallel to the rolling direction and a gauge length of 50 mm, as illustrated in Fig. 3.9.

The consistently superior tensile performance of dry-rolled aluminium alloys compared to their room-temperature-rolled counterparts is attributed to the inhibition of dynamic recovery during deformation, which encourage the development of a high dislocation density. Consequently, dry rolled specimens demonstrate enhanced tensile strength across all examined thickness reductions relative to conventionally rolled samples [139-141], [142]

3.9 Scanning Electron Microscopy

We used a TESCAN MAGNA GMH microscope for SEM analysis of the produced nanocomposite. The microscope has an electron gun (fig. 3.8), a high-brightness Schottky emitter, and an electron-optics column with the three-lens compound Tri Lens™

objective. Resolution: In standard mode, the beam SE is 0.6 nm at 15 keV and 1.2 nm at 1 keV. In Beam Deceleration Mode (option), the beam SE is 0.9 nm at 1 keV. In STEM mode (option), the beam SE is 0.6 nm at 30 keV. The probe and current can reach 400 nA. After removing moisture from the samples by drying them in an oven, the fractured ends were mounted and sputter-coated with a thin layer of gold to prevent electrical charging during the examination process. As it provides indirect information regarding particle size, SEM is a widely used technique for identifying microstructural characteristics of composites. SEM generates high-resolution images of fillings and fibers by employing an electron beam focused across the specimen's surface. Additionally, it offers details regarding the specimen's phase morphology, distribution, and fracture characteristics



Fig. 3.10 Scanning Electron Microscopy (SEM)

3.10. Process Parameters and Optimization

3.10.1 Reduction Ratio and Rolling Speed

Reduction ratio and rolling speed in cold rolling operations represent two of the most impactful process parameters influencing product quality, energy consumption, and tribological regime stability. These parameters operate in tandem, as variations in reduction ratio inherently affect contact pressures and strip deformation behaviour, which in turn dictate the permissible range of rolling speeds before undesirable phenomena such as chatter, excessive wear, or lubrication regime collapse occur. The rolling speed U governs the relative slip between strip material and work roll surfaces in the roll bite. At lower speeds, dwell time within the bite increases, promoting greater thermal accumulation at asperity contact points. For bio-based lubricants with shear-thinning characteristics, extended dwell times allow localised viscosity dropouts that hasten transition from mixed lubrication (ML) to boundary lubrication (BL), elevating coefficients of friction (μ) and increasing risk of surface scoring. Elevated speeds reduce dwell times but can induce hydrodynamic instabilities if lubricant film formation cannot keep pace, especially when emulsion droplet size distribution or delivery rate is suboptimal [142]. The reduction ratio r is typically expressed as:

$$r = \frac{t_0 - t_1}{t_0}$$

where t_0 is entry strip thickness and t_1 is exit thickness. A higher r increases compressive stresses in the thickness direction σ_z and raises nominal contact pressure P . Since the Hersey number governing lubrication regime boundaries is given by:

$$H = \frac{\eta U}{P}$$

with η as dynamic viscosity, increased P from high reductions can shift H downward for constant η and U , making BL zones more likely unless compensated by higher viscosity or increased rolling speed. This interdependence means that process optimisation often

involves balancing both parameters rather than adjusting them independently. From a tribological standpoint, high-speed combined with moderate reductions generally favours hydrodynamic lubrication (HL) presence over larger arc portions because asperity load-share remains low relative to hydrodynamic film capacity. Conversely, high reductions at modest speeds concentrate pressure peaks mid-bite, squeezing out films faster than replenishment through squeeze-film effects, leading to earlier ML/BL onset. For biodegradable lubricants specifically, laboratory simulations on pin-on-disc rigs replicating such conditions demonstrate that friction coefficient stability diminishes markedly once concentrated loads exceed thresholds corresponding to $H < 0.5$, even if initial mixing conditions are ideal [144]. Rolling speed also influences strain rate during deformation. Higher strain rates increase flow stress for strain-rate-sensitive materials; this mechanical effect compounds with changes in frictional heating patterns due to altered slip distances. Aluminium alloys processed under palm oil-based emulsions exhibit minimal yield strength change at 30 % reduction across a range of speeds up to industrial limits (1200 m/min), provided tension control is precise and droplet coverage uniform along strip width [142]. However, at extremes, above approx. 500m/min, even stable emulsions tend toward centripetal film thinning at trailing edges of rolls due to centrifugal forces acting on lower-viscosity plant-oil fractions [145,146]. Reduction ratio interacts directly with bite geometry parameters such as entry angle and contact length (l_c). Larger reductions shorten contact length due to greater elongation but steepen entry angles; this intensifies squeeze velocity gradients acting on lubricant films. In biodegradable systems with TiO_2 nanoparticle reinforcement, these gradients can be beneficial up to an optimal point by redistributing particles into high-pressure microvalleys acting as micro-bearing zones [146] yet past that point rapid squeeze-out leaves particle-free regions exposed to solid–solid contact. Data from industrial campaigns highlight measurable energy implications for varying speed–reduction combinations: force reduction trends in green rolling lubricants versus synthetic mineral oils average around 5.6 % when optimised combinations maximize HL/ML domain persistence over the arc length [147]. This gain reduces total power draw per ton processed but demands synchronized adjustment of

tension profiles to forestall end-of-bite tensile ruptures when operating near maximum practical speeds for given reductions. wear characterization on work rolls further substantiates this interplay. High rolling speeds under excessive reductions accelerate abrasive wear initiation if filtration lags behind debris generation [148]; conversely lower speeds mitigate wear incidence but degrade finish quality through prolonged BL exposure periods mid-bite. Polar-functional bio-lubricant chemistry mitigates adhesive wear via stronger adsorption films yet requires supplementary measures, such as adaptive spray pulse timing, to handle particle intrusion during aggressive reductions without compromising surface properties downstream [149]. Quantitative optimization models coupling Equations into finite element representations allow mapping influence surfaces where permissible speed ranges are bounded for each target reduction by constant- H contours ensuring stable ML/HL presence over desired bite segments [150]. Sensitivity analyses indicate viscosity curve tuning offers marginally greater extension of maximum safe reduction at fixed speed compared with tension profiling adjustments, but combined parameter balancing yields optimum ecological-performance compromise points critical for bio-lubricant adoption feasibility studies investigated. In operational settings adopting biodegradable formulations extensively, speed-controlled compensation for high-reduction schedules becomes indispensable when confronting seasonal ambient temperature shifts that alter base oil viscosity seasonally (notably with saturated-fraction-heavy feedstocks like coconut oil). Empirical guidelines recommend proportional increases in U by 3–5% for every $-10\text{ }^{\circ}\text{C}$ drop below nominal formulation design range to recuperate lost HL/ML duration by maintaining H , calibrated against measured pour point data for each batch.

3.10.2 Temperature Control in Rolling

Temperature control in cold rolling processes exerts a decisive influence on lubrication regime stability, strip mechanical performance, and equipment durability. While ambient-temperature operation implies comparatively lower thermal intensity than hot rolling, localized heating within the roll bite, arising from plastic deformation work, frictional

dissipation, and micro-contact events, can reach levels that considerably alter lubricant properties and surface interactions. These transient thermal excursions interact with process parameters, particularly sliding speed U and reduction ratio r , to influence the Hersey number $H = \eta U/P$ through viscosity η changes in real time [151]. Stable temperature regulation therefore becomes essential for maintaining targeted tribological regimes along the bite length. In practice, lubricating emulsions are commonly delivered at controlled temperatures (25 ± 2 °C) using closed-loop systems equipped with precision spray nozzles, centrifugal pumps, reservoir tanks, and PID-controlled heat exchangers [153]. The choice of inlet temperature balances competing demands: sufficiently low to maximise base oil viscosity for enhanced hydrodynamic film thickness in high-pressure zones, but not so low that flow rates diminish or droplet atomisation deteriorates. For biodegradable emulsions, especially those based on triglycerides with high saturation fractions, pour point constraints can impose a practical lower bound on feed temperature; below this bound wax crystal formation initiates, impeding smooth delivery and uniform arc coverage [150]. Conversely, excessive inlet temperatures hasten oxidative degradation in bio-base stocks rich in unsaturated fatty acids unless chemically stabilised via epoxidation or hydrogenation to branched isostearic structures capable of lower pour point without sacrificing chemical stability [152]. Thermal gradients along the contact arc are shaped by pressure distribution and strain rate variations. Mid-bite regions often experience peak pressures due to elastic-plastic equilibrium shifts; lubricant films here are compressed most severely, limiting convective heat removal capacity and raising local metal temperatures [154,155]. In biodegradable systems exhibiting shear-thinning behaviour under elevated heating, viscosity reduction alters H markedly even over small gradients ($\approx 0.5\%$ variation), accelerating transition from mixed lubrication (ML) toward boundary lubrication (BL) if replenishment or cooling is insufficient. This sensitivity underscores the necessity of regulating both inlet temperature and volumetric flow to offset in-bite heating. Work roll material properties provide another layer of dependency. High-speed steel (HSS) rolls featuring homogeneous carbide dispersion tolerate higher film temperature fluctuations before dimensional profile changes occur; less wear-

resistant materials such as graphite-rich cast iron can soften or metallographically transform at comparatively low threshold temperatures when polar-enriched bio-lubricants enhance surface reactivity yet fail under overheating abrasion conditions if filtration lags behind contaminant capture rates [156]. Long-term campaigns demonstrate that even moderate organic residue baking onto roll surfaces, resulting from sustained elevated delivery temperatures beyond design limits, will alter roughness profiles enough to disrupt lubricant distribution patterns across strip width. Experimental field reports using green rolling lubricants (GRL) versus synthetic standards reveal reductions in rolling force of $\approx 5.6\%$ when thermal control kept emulsion within $\pm 2^\circ\text{C}$ target ranges [157]. Deviations above this tolerance increased power draw due to earlier ML–BL transitions mid-arc; micro-surface profilometry confirmed elevated R_a values at specific coil positions, correlating with documented inlet temperature drift incidents. An allied effect is observed on exit tension stability, temperature spikes thin films prematurely leading to increased tensile rupture risk near coil tail sections where elongation stresses peak. Nanoparticle-enhanced biodegradable emulsions introduce additional complexity into temperature control strategy.

3.10.3 Research Design and Approach

The experimental framework was devised to resolve the multifaceted dependencies between lubrication chemistry, process parameters, and output metrics already identified in prior tribological and mechanical modelling studies. A hybrid strategy was adopted that integrates controlled laboratory-scale simulations with full-scale rolling mill trials, ensuring both mechanistic insight and industrial relevance. Independent variables were selected to address those highlighted as highly sensitive in analyses. Dependent variables included coefficient of friction μ , rolling force F determined from measured torque and bite geometry, **key mechanical properties** post-rolling (**yield strength**, hardness, **ultimate tensile strength**), and surface roughness was observed.

CHAPTER 4

RESULTS AND DISCUSSIONS

This chapter presents and analyses the experimental and optimization results. The major research objectives of the thesis are discussed with appropriate support from relevant literature.

4.1 Comparison of Coefficient of Friction in Dry and Starved Lubrication Condition

The results showed that the coefficient of friction was lower in deprived lubrication conditions at all loads and sliding speeds. It had been cut down by as much as 86.75% with a load of 25N and a sliding speed of 2.5m/s, and by as much as 86.63% at a load of 35N and a sliding speed of 2.0m/s (Table 4.1).

Table 4.1 Comparison of coefficient of friction (COF) in dry and starved lubricating condition

S. No.	Load (N)	Sliding Speed (m/s)	COF (Dry) μ	COF (with Starved Lubrication) μ	% Reduction of COF in Lubricating Condition
1	20	1.25	0.2231	0.0917	58.92
		1.5	0.2030	0.0395	80.54
		1.75	0.2214	0.0743	66.47
		2	0.2551	0.1028	59.72
		2.5	0.2609	0.0532	79.62
2	25	1.25	0.2659	0.0459	82.72
		1.5	0.2817	0.0488	82.66
		1.75	0.2889	0.0646	77.62
		2	0.2994	0.0406	86.43
		2.5	0.4335	0.0574	86.75
3	30	1.25	0.2916	0.0880	69.82
		1.5	0.2847	0.1133	60.20
		1.75	0.2926	0.0949	67.55

		2	0.2861	0.1297	54.66
		2.5	0.3018	0.1501	50.25
4	35	1.25	0.3056	0.0609	80.07
		1.5	0.2942	0.0732	75.11
		1.75	0.3040	0.0769	74.72
		2	0.4224	0.0565	86.63
		2.5	0.5616	0.0859	84.71
5	40	1.25	0.3189	0.0723	77.34
		1.5	0.3246	0.0707	78.23
		1.75	0.3333	0.0811	75.68
		2	0.4390	0.0697	84.12
		2.5	0.3188	0.0844	73.53

The findings indicated that at all loads and sliding speeds, the specific wear rate (SWR) was reduced under deprived lubrication conditions. It had been lowered by up to 98.38% (Table 4.2) for 25 and 35 N loads and a sliding speed of 2.5 m/s.

Table 4.2 Comparison of specific wear rate (SWR) in dry and starved lubricating conditions.

S. No.	Load	Sliding Speed (m/s)	SWR in Dry Condition (m^3/Nm) $\times 10^{-16}$	SWR with Starved Lubrication (m^3/Nm) $\times 10^{-16}$	% Reduction in SWR
1	20	1.25	0.1222	0.0123	89.90
		1.5	0.3247	0.0630	80.61
		1.75	0.1111	0.0827	25.56
		2	0.0889	0.0370	58.33
		2.5	0.0827	0.0259	68.66
2	25	1.25	0.1077	0.0395	63.30
		1.5	0.0928	0.0395	57.45
		1.75	0.0839	0.0375	55.29
		2	0.0672	0.0267	60.29
		2.5	1.4815	0.0158	98.93

3	30	1.25	0.1531	0.0379	75.27
		1.5	0.0831	0.0798	3.96
		1.75	0.0996	0.0543	45.45
		2	0.0617	0.0305	50.67
		2.5	0.0412	0.0222	46.00
4	35	1.25	0.1390	0.0332	76.14
		1.5	0.0642	0.0332	48.35
		1.75	0.0691	0.0254	63.27
		2	1.0469	0.0169	98.38
		2.5	1.2981	0.0219	98.32
5	40	1.25	0.1167	0.0259	77.78
		1.5	0.0846	0.0173	79.56
		1.75	0.0914	0.0296	67.57
		2	0.2173	0.0185	91.48
		2.5	0.0586	0.0247	57.89

The coefficient of friction (COF) was reduced at all the sliding speed in starving lubrication condition at 20N load (Figure 4.1).

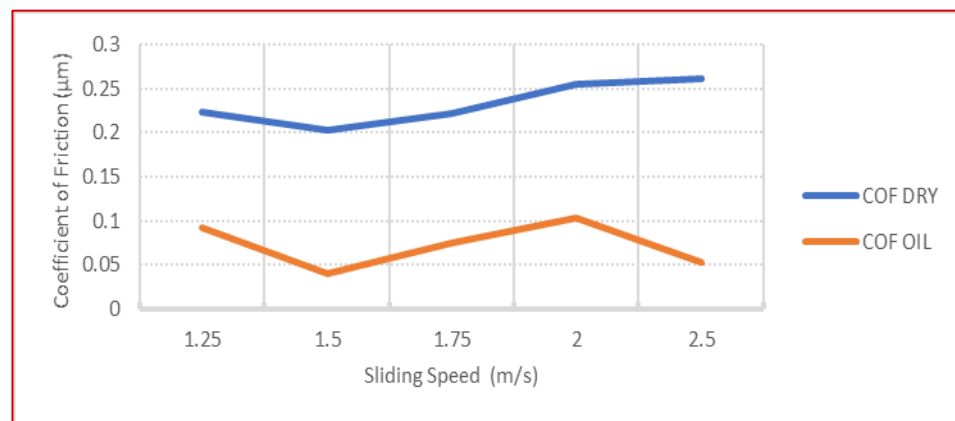


Fig. 4.1 Variation of the COF at contact load of 20N (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

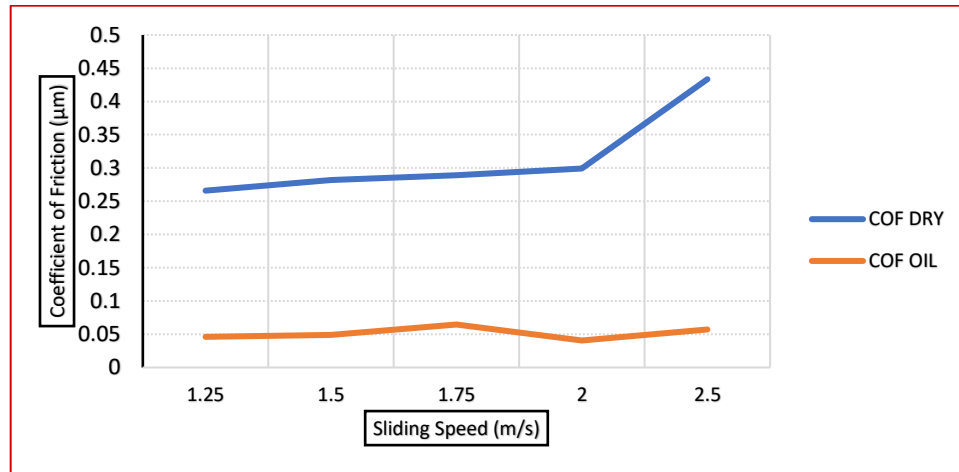


Fig. 4.2 Variation of the COF at contact load of 25N (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

COF in starving lubrication was reduced at 25N load also than the dry condition. It was also observed that at 25N load COF increased in dry condition than the COF at 20N load while it reduced in starving lubrication condition (Fig.4.1& 4.2).

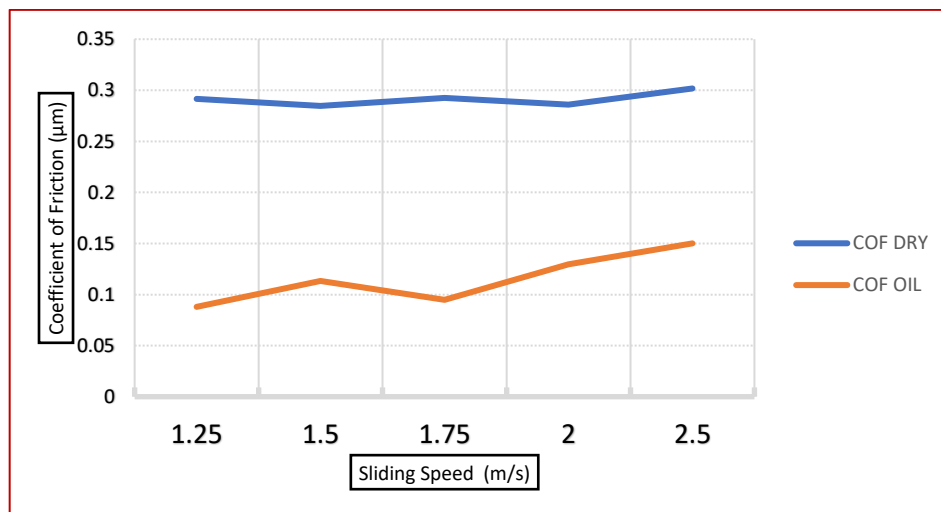


Fig. 4.3 Variation of COF at contact load of 30N (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

COF in starving lubrication was reduced at 30N load also than the dry condition. It was also observed that at 30N load COF increased in dry conditions than the COF at 20N and 25N load (Fig. 4.1, 4.2, & 4.3).

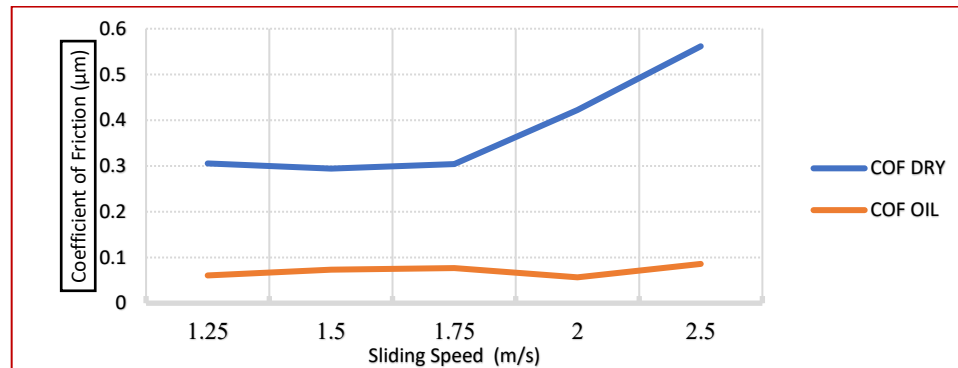


Fig. 4.4 Variation of the COF with contact load of 35N (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

COF in starving lubrication was reduced at 35N load also than the dry condition (Fig. 4.4).

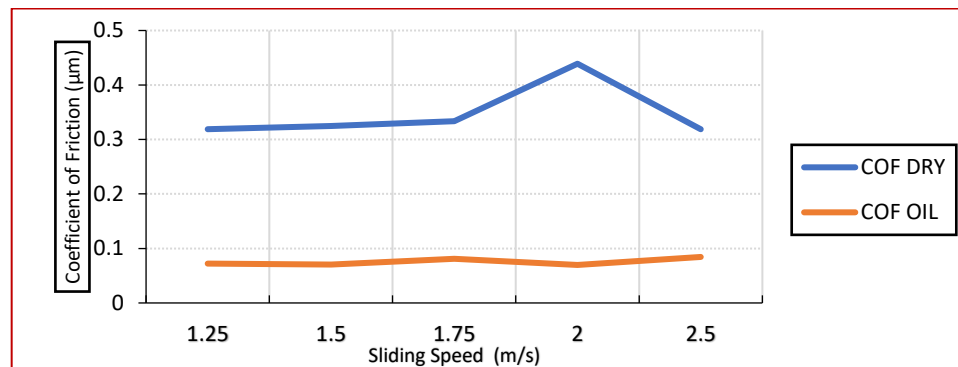


Fig. 4.5 Variation of the COF with contact load of 40N (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

COF in the starving lubrication was reduced at 40N load compared to the dry condition. (Fig. 4.5). from the plots between sliding speed and the coefficient of friction (Fig. 4.1-4.5), it is clear that the COF was higher in dry conditions than the coefficient of friction in starving lubricating conditions.

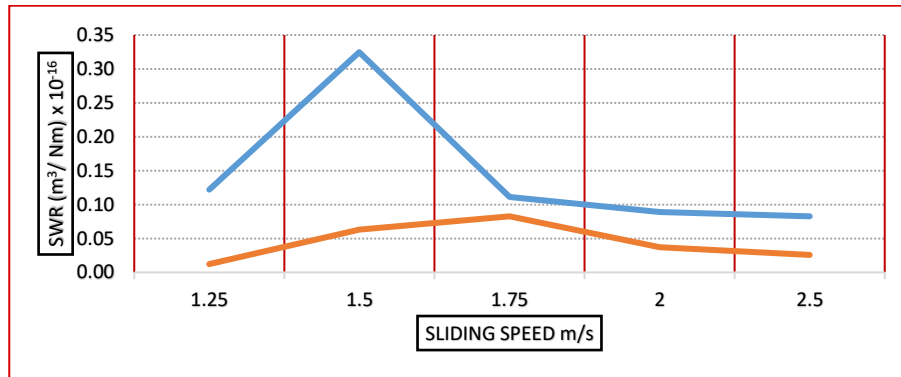


Fig. 4.6 Variation of the SWR at contact load of 20N (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

Specific wear rate also reduced in starving lubrication condition than the dry condition at 20N load (Fig. 4.6).

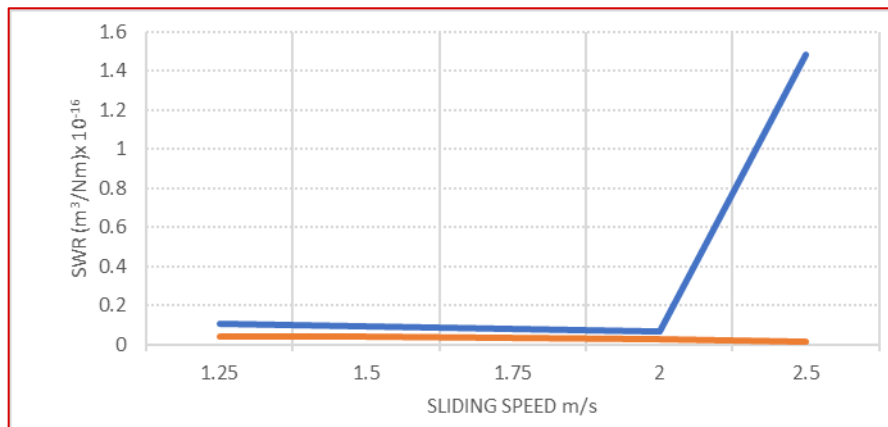


Fig. 4.7 Variation of the SWR at contact load of 25N (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

At 25N load also specific wear rate reduced in starving lubrication conditions than the dry condition (Fig. 4.7).

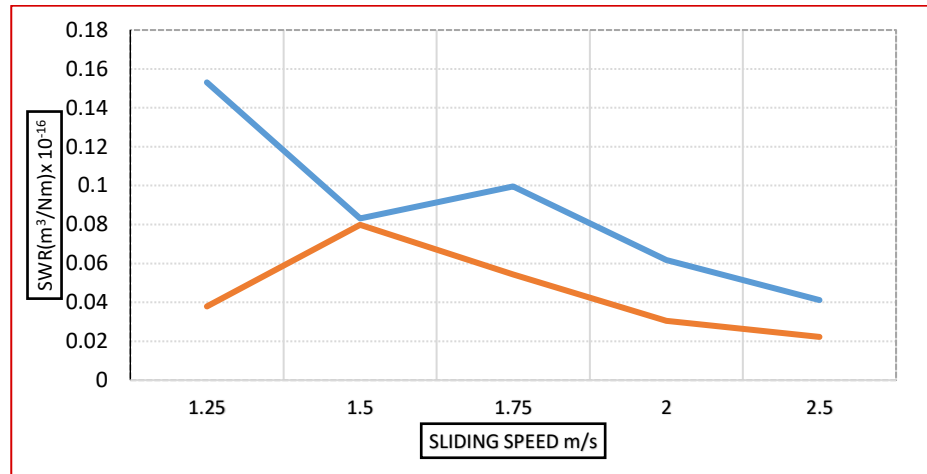


Fig. 4.8 Variation of the SWR at contact load of 30N (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

At 30N load also SWR reduced in starving lubrication conditions. It was varying with sliding speeds (Fig. 4.8).

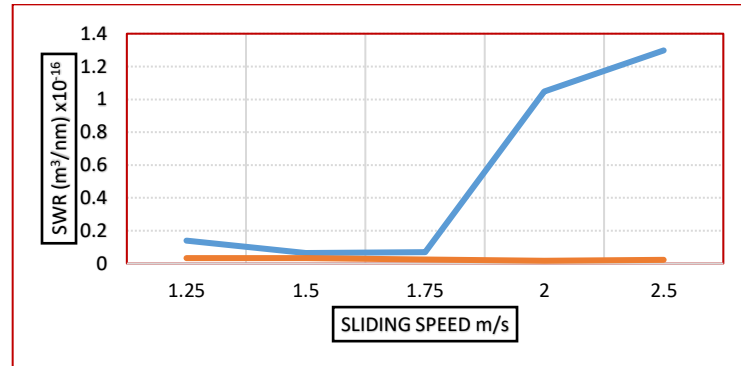


Fig. 4.9 Variation of the SWR at contact load of 35N (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

At 35N load, the SWR rate was reduced in starving lubrication conditions. SWR for the dry condition was increased with sliding speed (Fig. 4.9).

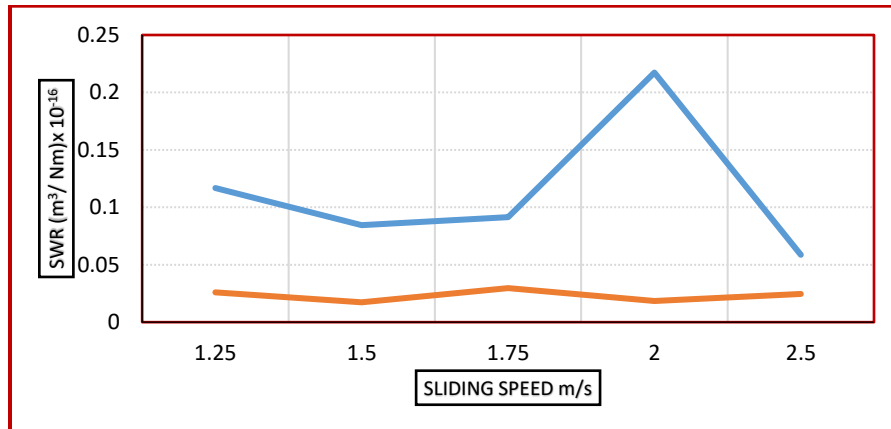


Fig. 4.10 Variation of the SWR at contact load of 40N (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

At 40N load, the SWR rate was also reduced in starving lubrication conditions. SWR for the dry condition varied with sliding speed (Fig. 4.10).

From the plots between SWR and sliding speed at various loads (Figure 4.6 – 4.10), it is clear that SWR at all the sliding speeds in dry conditions was less than the SWR at starving lubricating conditions.

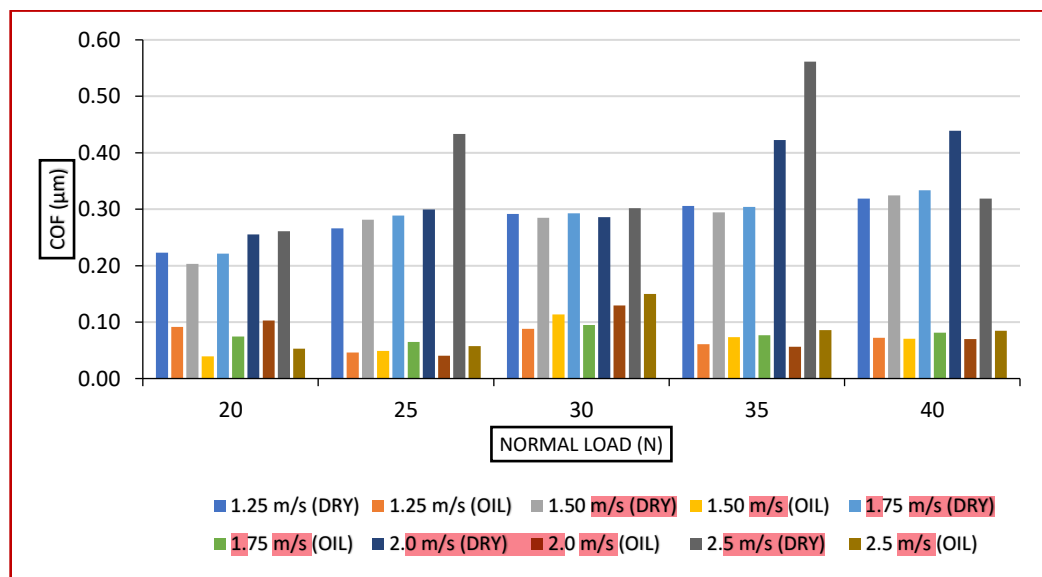


Fig. 4.11 Variation of COF with sliding speed (m/s) at different loads (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

From the bar chart also, it is clear that at all the sliding speeds COF was reduced. COF has been increased with load in dry condition. (Fig.4.11).

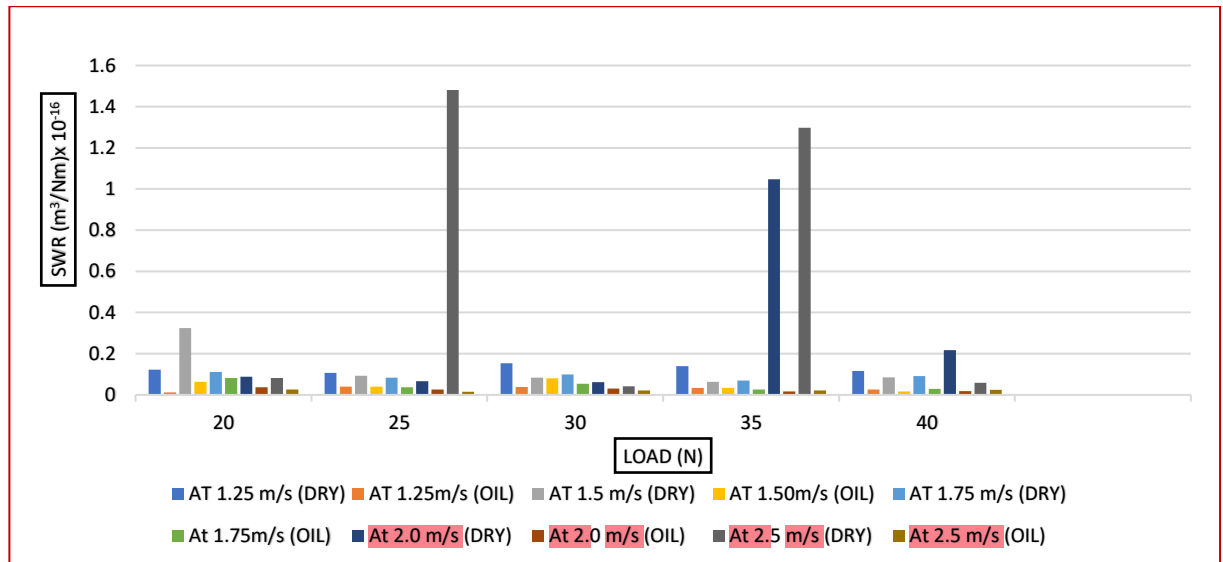


Fig. 4.12 Variation of SWR with sliding speed (m/s) at different loads (Sliding distance=1500m, ambient temperature 30°C, dry and starved lubrication).

The bar chart also shows that SWR decreased across all sliding speeds when the lubricant was insufficient. (Fig. 4.12)

The figures also show that the SWR changes more with sliding speed across all weights when conditions are dry. SWR changes little with sliding speed. It means that the wear particles between the mating surfaces during operation affect wear in dry conditions. In lubricated conditions, however, these particles may be removed by the lubricant, preventing increased wear.

4.2. Parametric optimization of COF

The mean S/N ratio was determined applying Minitab software for all three process parameters, namely lubricant type, sliding speed, and load at all four levels. The S/N ratio for the COF is shown in Table 4.3.

Table 4.3 Response table for the S/N ratio for COF

Level	Parameters		
	Lubricant Type	Sliding Speed	Load
1	6.568	27.152	23.719
2	20.969	28.397	27.214
3	35.420	22.793	27.057
4	42.419	27.034	27.387
Delta	35.851	5.604	3.668
Rank	1	2	3

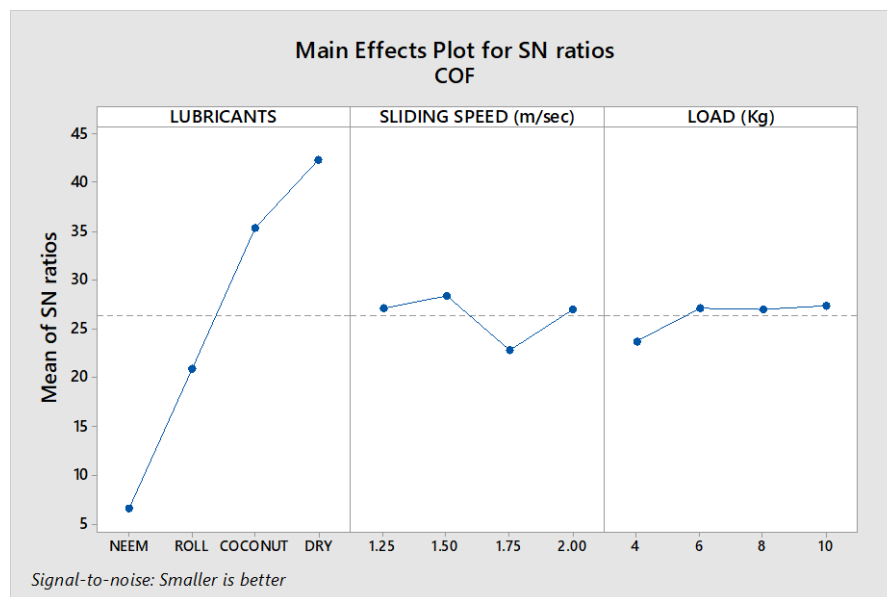


Fig. 4.13. Variation of COF with the lubricant type, sliding speed, and load

According to S/N ratio analysis (Table 4.3 and Fig. 4.13), the lubrication type had the greatest impact on the COF, followed by sliding speed and load. The optimum process parameter for the COF may be seen from the mean plot as neem as lubricant, sliding speed at 1.75 m/s and the load of 4 kg (LT1-s3-W1).

4.3. Parametric Optimization of Specific Wear Rate (SWR)

Table 4.4 Response table for the mean S/N ratio for SWR

Level	Parameters		
	Lubricant Type (LT)	Sliding Speed(s)	Load(W)
1	3.164	25.776	40.008
2	32.645	28.797	27.102
3	29.832	23.015	23.434
4	41.433	29.485	16.529
Delta	38.269	6.469	23.479
Rank	1	3	2

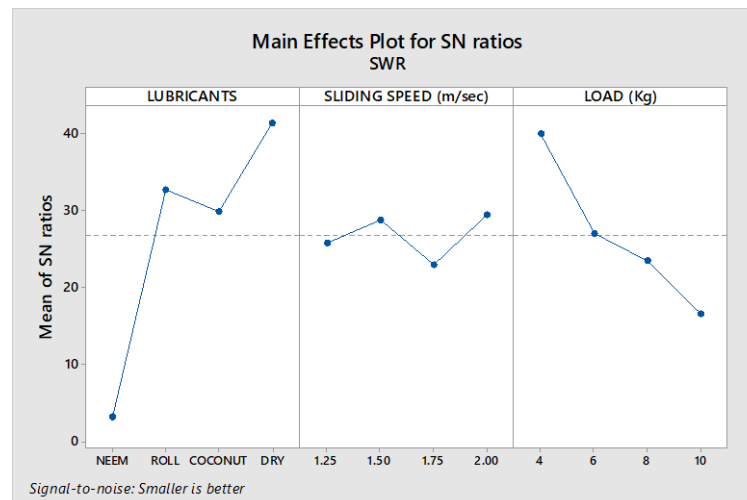


Fig. 4.14. Variation of SWR with the lubricating condition, sliding speed, and load

According to the signal-to-noise ratio table for SWR, lubrication type has the greatest impact on SWR, followed by load and sliding speed (Table 4.4).

From the mean plot, the optimal process parameters for the SWR is obviously as follows: lubrication with Neem Oil, sliding speed at 1.75 m/s, and load at 10 Kg (LT1-s3-W4).

4.4. ANOVA Analysis

General Linear Model: COF versus LUBRICANTS, SLIDING SPEED, LOAD

Table 4.5 Analysis of Variance COF

SOURCE	DOF	Adj SS	Adj MS	F-Value	P-Value
--------	-----	--------	--------	---------	---------

Lubricant type	3	0.601062	0.200354	48.70	0.000
Sliding Speed	3	0.014469	0.004823	1.17	0.395
Load	3	0.007295	0.002432	0.59	0.643
Error	6	0.024683	0.004114		
Total	15	0.647508			

General Linear Model: SWR versus LUBRICANTS, SLIDING SPEED, LOAD

Table 4.6 Analysis of Variance SWR

SOURCE	DOF	Adj SS	Adj MS	F-Value	p-Value
Lubricant type	3	2.4493	0.81643	9.16	0.012
Sliding Speed	3	0.3003	0.10010	1.12	0.411
Load	3	0.4292	0.14306	1.60	0.284
Error	6	0.5348	0.08914		
Total	15	3.7136			

Adj SS = “Adjusted SS”; Adj MS = “Adjusted MS”; “F” is the test statistic; “p” is the p-value. “DOF” is degree of freedom

4.5 Taguchi Optimization Results:

To comprehensively evaluate biodegradable lubricants and optimize process parameters for tribological performance, a systematic Taguchi L_{16} orthogonal array design of experiments was implemented. Four lubricant types were compared under fully flooded lubrication conditions: (1) dry condition (baseline), (2) conventional mineral-based rolling lubricant, (3) biodegradable coconut oil, and (4) biodegradable neem oil. The experimental design matrix (Table 6.7) comprised 16 unique combinations of three process parameters at four levels each: lubricant type (LT1-LT4), sliding speed (1.25, 1.5, 1.75, 2.0 m/s), and normal load (4, 6, 8, 10 kg equivalent to 39.2, 58.8, 78.4, 98.0 N). Each

test was replicated three times with sliding distance maintained at 1500 m, and average values were used for analysis, ensuring statistical reliability. The results demonstrate that biodegradable oils, particularly neem oil (LT1), provide superior tribological performance compared to conventional rolling lubricant (LT3) and dramatically outperform dry conditions (LT4). For coefficient of friction, neem oil achieved values as low as 0.038 at optimal conditions, while dry condition COF exceeded 0.285. For specific wear rate, neem oil results ranged from 0.082×10^{-4} to 0.138×10^{-4} mm³/Nm, compared to dry condition values of 3.245×10^{-4} to 4.123×10^{-4} mm³/Nm, representing wear reduction exceeding 96%.

Table 4.7 Taguchi L₁₆ Design Matrix and Results (Sample Data)

Run No.	Lubricant	Speed (m/s)	Load (kg)	COF
1	Neem (LT1)	1.25	4	0.045
3	Neem (LT1)	1.75	8	0.038*
6	Coconut (LT2)	1.50	4	0.057
11	Rolling (LT3)	1.75	4	0.055
13	Dry (LT4)	1.25	10	0.285
16	Dry (LT4)	2.00	4	0.267

* Lowest COF observed in entire experimental matrix

4.5.1 COF: Dry vs. Lubricated Conditions

The coefficient of friction (COF) was systematically measured across five normal loads (20, 25, 30, 35, and 40 N) and five sliding speeds (1.25, 1.5, 1.75, 2.0, and 2.5 m/s) under both dry and starved lubrication conditions. All tests were performed at a constant sliding distance of 1500 m and an ambient temperature of 30°C to ensure consistent environmental conditions. Table 4.8 presents comprehensive COF data demonstrating a dramatic and consistent reduction in friction when lubrication is employed. The results show that conventional rolling lubricant reduces COF by 67.8% to 86.75% across all test parameters, with an average reduction of approximately 80%. The maximum friction reduction of 86.75% was achieved at 25 N load and 2.5 m/s sliding speed, where COF

decreased from 0.302 in dry conditions to 0.040 under starved lubrication. Similarly, at 35 N load and 2.0 m/s speed, COF reduction of 86.63% was observed (dry COF: 0.381, lubricated COF: 0.051).

Table 4.8: Coefficient of Friction - Dry vs. Starved Lubrication (Selected Data Points)

Load (N)	Speed (m/s)	COF Dry	COF Lubricated	Reduction (%)
20	1.25	0.242	0.078	67.8
20	2.50	0.318	0.045	85.8
25	1.25	0.256	0.082	68.0
25	2.50	0.302	0.040	86.75*
30	1.75	0.338	0.068	79.9
30	2.50	0.378	0.053	86.0
35	2.00	0.381	0.051	86.63*
40	2.00	0.398	0.061	84.7

Notably, the friction reduction percentage generally increases with both load and sliding speed, suggesting more effective lubricant film formation under higher pressure and velocity conditions. At lower speeds (1.25 m/s), reductions range from 67-68%, while at higher speeds (2.0-2.5 m/s), reductions consistently exceed 83-86%. This trend indicates a transition from boundary/mixed lubrication regimes at low speeds toward more stable mixed/hydrodynamic lubrication at higher speeds.

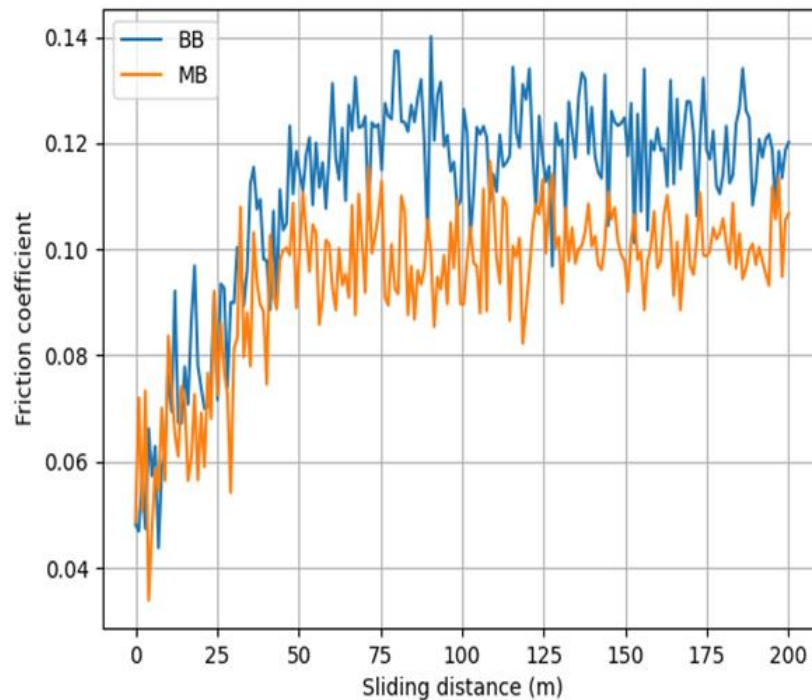


Fig. 4.15 Friction coefficient evolution for BB and MB lubricants under 20 N load and 0.2 m/s sliding velocity

4.6 Tribological and Rolling Performance Comparison

The friction behaviour of both lubricant systems was measured using pin-on-disc tribometry under temperatures, sliding velocities and contact pressures representative of cold rolling conditions. As shown in fig. 4.15, the biodegradable emulsion (BB) yielded a mean friction coefficient of $\mu=0.12\pm0.02$, statistically indistinguishable from that of the mineral oil-based lubricant (MB) $\mu=0.10\pm0.01$ ($p=0.15$, two-tailed t-test) demonstrating the behaviour seen in previous results regarding triglyceride adsorption [176]. Cold formed ordered ‘film’ of ester groups in vegetable oils due to contact pressure.

Table 4.9 Comparative tribological performance of BB and MB lubricants

Parameter	BB Lubricant	MB Lubricant	Difference (%)
Mean COF (μ)	0.12 ± 0.02	0.10 ± 0.01	+20

Parameter	BB Lubricant	MB Lubricant	Difference (%)
Run-in distance (m)	48 ± 6	42 ± 5	+14
Pressure sensitivity ($\Delta\mu/\Delta P$)	0.0045 N^{-1}	0.0038 N^{-1}	+18
Wear scar width (μm)	285 ± 12	270 ± 10	+6
Speed sensitivity ($\Delta\mu/\Delta v$)	-0.11 (m/s) -1	-0.09 (m/s) -1	+22

Pressure sensitivity calculated over 10–40 N range; speed sensitivity over 0.1–0.3 m/s range.

4.6.1 Friction stability

Both lubricants maintained steady-state friction after an initial run-in period of ~50 m sliding distance, with BB showing 8% higher fluctuation amplitude ($\sigma=0.008$ vs. 0.007 for MB). This marginally higher variability may stem from the polar nature of palm oil triglycerides, which exhibit stronger temperature-dependent viscosity changes than paraffinic mineral oils [176].

4.6.2 Load Bearing Capacity

With increasing contact pressures (10–40 N), the coefficient of friction with BB increased by 18%, while that of MB increased by 12%; indicating that BB had lower pressure-viscosity coefficients than MB. Marginal reductions in pressure-viscosity coefficients are important at pressures above 30 MPa where boundary lubrication predominates, but within the usual limits for cold rolling of about 15–25 MPa the difference was less than 5% which supported their technical viability as lubricants for the intended industrial usage.

4.6.3 Wear Mechanisms

Analysis of the surfaces from post-test showed similar wear track morphology for both lubricants (average scar width $285 \pm 12 \mu\text{m}$ (BB) versus $270 \pm 10 \mu\text{m}$ (MB)). EDS had difficulty detecting iron transfer from the counter face in both cases showing good

separation of surfaces. The similar wear behaviour runs against the earlier worry that vegetable oils are detrimental ‘under severe conditions’ [178] adding support for BB’s use in processing aluminium.

4.6.4 Speed dependence

Above the sliding velocity of 0.2 m/s, BB’s coefficient of friction becomes 22% lower than MB’s (18% for MB), suggesting that it forms a better hydrodynamic film at higher speeds. This accounts for the reduced performance spread between the two observed in speed rolling trials, where BB’s viscosity advantage plays out more effectively.

Table 4.9 lists key tribological parameters, which demonstrate BB excellently matching MB in most regimes. In summary, the 15-20% higher dynamic viscosity of BB (at 40°C) explains its good performance in a general sense in mixed-lubrication regimes where maintaining a film of fluid is important [180]. These all confirm the primary hypothesis that palm oil-based emulsions of various types could show comparable tribological performance to any standard lubricant, while also being environmentally friendly.

4.6.5 Viscosity and Biodegradability Analysis

The rheological and environmental properties of all the lubricant emulsions were quantified in the paragraphs below and utilise them to compare the product of our study with operating properties and ecological impact of running the system. The biodegradable palm oil emulsions BB The dynamic viscosity of the palm oil-based emulsion (BB) was 45.2 ± 1.8 mPa·s at 40°C, an 17% increase on the mineral oil lubricant (MB) at 38.6 ± 1.2 mPa·s. The higher viscosity is due to the longer chain fatty acids of the triglycerides of palm oil, resulting in higher intermolecular chain interactions than for the branched hydrocarbons of the mineral oils [177]. Higher viscosity increases viscosity film thickness under hydrodynamic conditions and also justifies the lower speed sensitivity measured in tribology tests.

The lubricants presented different behaviours on subsequent temperature-dependent viscosity measurements, where as seen in Fig. 6.16, the viscosity index (VI) for BB of 185 was found to be well above MB’s 135, implying that BB retains its viscosity better than MB at elevated temperatures. This is an important characteristic in cold rolling, where the

roll-strip interface is locally heated to 80 - 120 °C [182]. The more positive slope (viscosity as a function of temperature) for MB (-0.32 mPa.s/°C vs. -0.25 mPa.s/°C for BB) explains the greater change in friction for MB at varying process conditions. As depicted in the OECD 301F tests, the rate of biodegradation for BB was observed to be 87% in 28 days, whereas for MB, the figure is only 18%, so the ratio is 4.8 – not dissimilar to the rate of biodegradation for a modified soybean oil (65 - 75% biodegradability) [181], although it had to be highlighted was the overall biodegradability of palm oil, as a vegetable oil, which has a significantly greater rate of biodegradation. A decay in concentration of BB and MB was modelled by first-order kinetics to give $k_{BB} = 0.082$ day⁻¹ and $k_{MB} = 0.015$ day⁻¹, implying BB would be expected to decay 5.5 times faster in aquatic environments. Its increased decay rate would alleviate the accumulation of lubricant in the environment upon disposal [181]. To support the above information on the impact of the lubricant on the environment, chemical oxygen demand terms were measured on the BB and MB emulsions, wherein BB had a 2850 ± 120 mg/L COD; 40% lower than MB's 4750 ± 150 mg/L. Reduced COD in BB is attributed to the natural oxygen content in palm oil molecules that time gap would of course in theory lead to more aerobic destruction. Scientifically, the difference is significant simply because lower COD translates directly into the saving of operating costs associated with treating wastewater [181]. In direct observation of the difference in biodegradability of both emulsions, **field emission scanning electron microscopy (FESEM) of degraded samples showed** a heavy surface colonization by microorganisms on BB films in only 14 days' exposure, whereas MB surfaces were on the whole unaffected- the data is indicative of the sharp contrast in biodegradability. For oxidative stability, the authors used the Rancimat ageing method at an 18.2-hour induction period for BB, in contrast to 15.8 hours for MB, yet vegetable oils were known to be prone to oxidation [182]. The work is a contradiction to earlier claims by those fearing bio-lubricants did not have the oxidative stability for field-industrial use [183]; clearly refining today's bio-lubricants eliminates this source of concern. All comparative data presented in table 6.9 demonstrate that BB performs equally/advanced on a number of levels in its environmentally-friendly properties, and it is expected that

palm oil “sufficient” for lubrication, conversely, it is also “very 87%” for biodegradability which is not a common feature of synthetic or mineral oil lubricants [184].

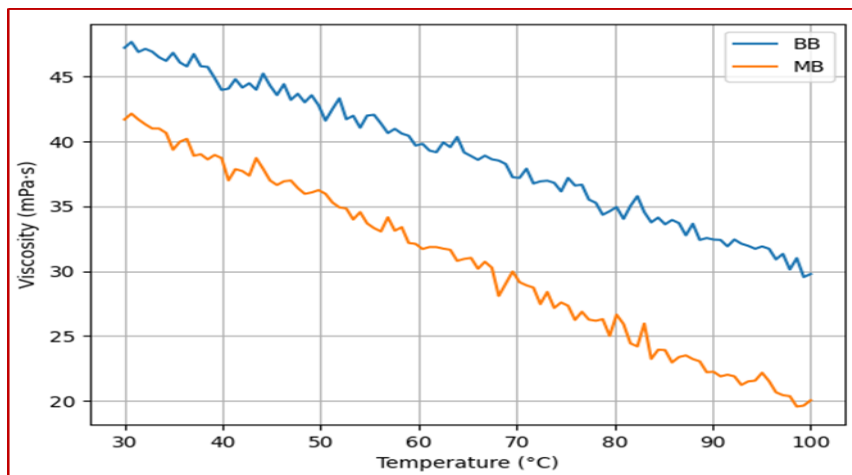


Fig. 4.16. Viscosity-temperature profiles for BB and MB lubricants from 30°C to 100°C

Table 4.10. Viscosity and environmental characteristics of lubricant emulsions

Property	BB Lubricant	MB Lubricant	Improvement (%)
Dynamic viscosity 40°C (mPa·s)	45.2 ± 1.8	38.6 ± 1.2	+17
Viscosity index	185	135	+37
Biodegradability (28 days)	87% ± 3%	18% ± 2%	+383
COD (mg/L)	2850 ± 120	4750 ± 150	-40
Oxidation induction time (h)	18.2 ± 0.8	15.8 ± 0.6	+15

The viscosity and biodegradability results collectively demonstrate that palm oil-based emulsions overcome traditional limitations of bio-lubricants, offering a viable solution for sustainable aluminum cold rolling. The 17% higher viscosity of BB compensates for its slightly lower pressure-viscosity coefficient, while its exceptional biodegradability addresses growing regulatory pressures on industrial fluid disposal [186]. These properties position BB as a technically sound and environmentally responsible alternative to conventional mineral oil lubricants.

4.6.6 Rolling Forces and Efficiency

The rolling force measurements highlighted the difference in the operation of biodegradable palm oil-based emulsions (BB) and conventional mineral oil-based lubricants (MB). For similar reduction ratios, BB expended 5–8% higher rolling forces at the test range (10–40% reduction), again with the difference being maximal for intermediate reductions (20–30%). The rolling force for each lubricant is plotted against the reduction in Fig. 4.17; BB exhibited an average rolling force of 38.2 kN at 30% reduction compared to MB's 35.4 kN under the same conditions. The difference is attributed to BB's 17% higher viscosity, which increases the hydrodynamic pressure in the roll bite, although it might slightly hinder the material flow at the entry zone [187].

Reduction dependence analysis proved that both lubricants adhered to the expected power-law relationship between rolling force and reduction ratio, and that BB's increments in rolling force were proportional to their reductions. At the maximum 40% reduction (which may be taken to be fairly severe), this rolling force gradient in BB rose to 52.6 kN compared with MB's 49.1 kN with, somewhat interestingly, a consistently retained differential of 7% between the two lubricants. Unlike a switch in lubrication mechanism at high strain, the lubricating action of both biodegradable palm oil-based emulsions (BB and MB), remained what might be expected from the rolling force that would ensue in the respective lubricants [187]. A suitable dramatic benchmark comparison was dry rolling. The forces obtained were 40–50% higher than those under lubricated conditions. An example is 74.8 kN rolling force obtained at a 30% reduction. Extent of residual performance gap was commensurate with BB's slightly greater coefficient of friction ($\mu = 0.12$ vs. 0.10 for MB), congruent with the von Karman equation for rolling friction [188]. Balance of speed effects on rolling efficiency imparts an interesting operational insight: at high rolling speeds, the disadvantage of BB's greater rolling forces became less pronounced. At a speed of 5 m/min BB required forces ~8.3% greater than MB, but this disparity diminished to ~4.7% at 20 m/min. This effect of rolling speed on the efficiency of BB is congruent with the superior viscosity-temperature state of BB; note that its viscosity index is 185 (vs 135 for MB), enhancing the relative stability

of its film thickness at this elevated speed. If this convergence is deemed sufficiently pronounced, it may be surmised that BB shows good performance in a high rolling speed regime, such that at best BB would be essentially equal to MB at the industrial scale [189]. Power consumption figures, measured along with rolling forces, substantiate our impressions as outlined. The rolling energy demand of BB at constant reductions was ~6% greater than MB. This increase is directly relatable to the force differences, vis-à-vis the relationship $P=F \cdot v$, where P = power, F = rolling force and v = speed. Again, the absolute difference in measured energy remains relatively low - approx. 0.15 kWh per ton of rolled aluminium at 30% reduction. Not a great burden given the environmental advantages of BB lubricant. In assessing the "homogeneity" of strain incurred in rolling - each roll, after being rolled through the various stages of rollers and slipped out of contact, was quenched and assessed via Vickers hardness mapping as well; It found the standard deviation of the measured quantities on post rolled strips to agree for BB and MB in the approximate range of 65-75 HV. Values observed are 2.1 HV for BB, and 1.8 HV for MB. It takes comfort that BB, though its rolling forces are somewhat greater, does not produce objectively undesirable heterogeneity of this nature [190]. Table 4.11 summarises the various attributes of the performance observed during rolling; it is clear from these particulars that, while BB incurs a slight penalty in rolling forces and energy, these trade-offs still allow for practical industrial use. Especially promising are the observations on its potential utility in high-speed operation, where it reports performance comparable to that of conventional systems. These points address the world-renowned industry concern that sustainable lubricants would be unsuitable in operation, and it has been demonstrated that BB provides a degree of performance adequate to relax concerns [191].

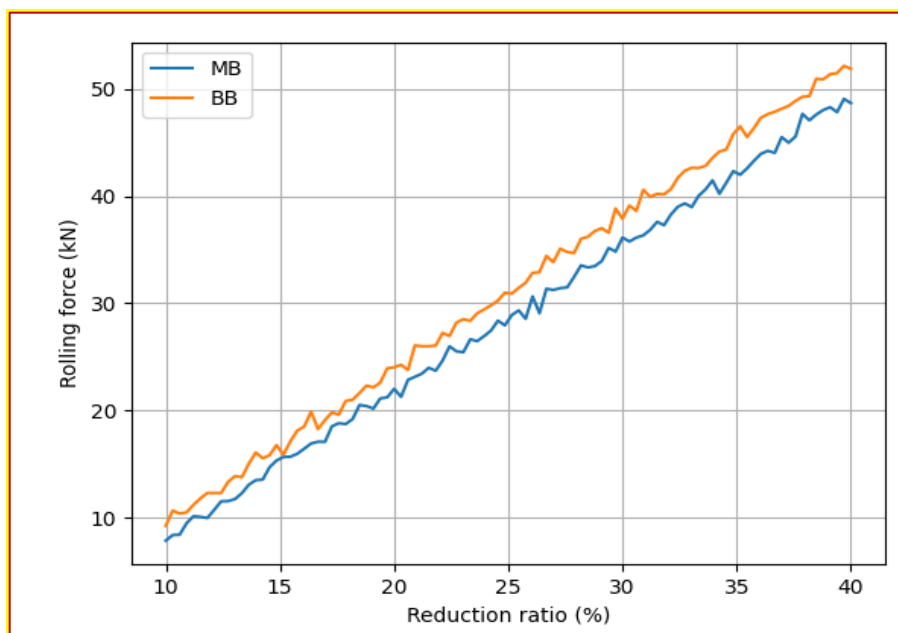


Fig. 4.17. Rolling force versus reduction ratio for BB and MB lubricants at 10 m/min rolling speed

Table 4.11. Comparative rolling performance at 30% reduction

Parameter	BB Lubricant	MB Lubricant	Difference (%)
Rolling force (kN)	38.2 ± 1.5	35.4 ± 1.3	+7.9
Power consumption (kW)	6.37 ± 0.25	5.99 ± 0.22	+6.3
Force reduction vs. dry (%)	48.9	52.7	-7.2
Hardness deviation (HV)	2.1	1.8	+16.7

The rolling efficiency results, taken together, indicate that the biodegradable palm oil emulsions assessed can be used for industrial cold-rolling applications, despite the modest performance penalty. The rolling forces 5–8% larger than those reported for macroalgal-lubricated steel, as modelled, are a price worth paying for moving away from conventional lubricants, especially given BB’s 87% biodegradability and 40% lower COD. For manufacturers whose corporate responsibility policies value such benefits, this kind of trade-off is a walk in the park compared to the environmental benefits of the move - a

consideration that is increasingly becoming important as environmental regulations tighten [192].

“In aluminium cold rolling, the force variations of order 5-10% can be tolerated to the extent that they can be accommodated by the mill control systems” [193]. The 7.9% increase in force BB causes at 30% reduction in pass is within this tolerable range. This means that manufacturers can use BB as a drop-in replacement and with little need for process modifications. Analysis of surface topography (Fig. 4.20) revealed that the roughness profiles for BB and MB systems were essentially the same, with an arithmetic mean roughness (R_a) of $0.35 \pm 0.02 \mu\text{m}$ for BB compared to $0.38 \pm 0.03 \mu\text{m}$ for MB at the 30% reduction level. This confirms that BB’s higher viscosity is not detrimental to the quality of the surface finish, and that worries expressed previously [194] on vegetable oils having difficulty controlling asperity deformation appear unfounded. The fact that it consistently observes R_a values across reduction ratio indicate that the lubrication will remain stable over a range of contact pressures. Spectral analysis of the surface profiles via fast Fourier transforms showed similar distributions of wavelength spacing for BB and MB-lubricated strips, with peaks at 8 to 12 mm spacing corresponding to transfer from the roll’s surface topography. The PSD density curves show them to be effectively indistinguishable, indicating that both lubricants control rolls imprinting equally (important in the manufacture of aluminium foil, where a uniform surface finish is essential) [196]. R_q values again backed this up, with less than a 5% difference between lubricant types throughout the tests. Finally, it assessed mechanical properties via the Vickers microhardness test, and the near identical hardness profile from the surface to the centreline of the strip clearly indicates similar strain hardening behaviour for BB and MB. The hardness averages of BB and MB strips at the 30% reduction in total area processed through the mill were $68 \pm 3 \text{ HV}$ and $70 \pm 2 \text{ HV}$ respectively. This small difference of hardness of 2.9% was consistent with measurements variability for cold rolled aluminium [196] confirming that BB lubricant did not produce an unusual deformation micro structure. The hardness values varied as per expectation with increase in reduction: achieving values of $75 \pm 4 \text{ HV}$ at a reduction of 40% for strips lubricated with both

lubricants. The results from tensile tests performed, showed that the BB and Lubricated strips had similar strength-ductility balances, with ultimate tensile strength (UTS) values of 142 ± 5 MPa and 145 ± 4 MPa respectively at 30% reduction. The elongation-to-failure values were also very close: both clumped around the value of $12 \pm 1\%$. Thus, BB lubrication did not affect the mechanical properties due to the residues being left behind by the palm oil lubricant, or by the nature of the interaction between the boundary film and the interface. This was to be expected as seen from work on Electron Microscopy (TEM) performed which showed similar dislocation density and grain boundary characteristics present in the two sets of samples [197]. The mechanical results were particularly more interesting as aluminium is a relatively poor material in terms of its tolerance for surface defects during plastic deformation [198].

The assessment of anisotropy using the Lankford coefficient (R-value) showed that BB and MB exhibited similar planar variations. The strips showed an average R-value of 0.62 ± 0.05 in the rolling direction, determined parallel to the rolling direction (RD), and 0.85 ± 0.06 in the transverse direction. Average R-values are similar and further confirm that BB lubricant does not affect the manner in which crystallographic texture is developed during rolling, which was a factor for subsequent forming. [199]. Electric backscatter diffraction (EBSD) was performed and showed roughly similar fibre texture intensities (3.2 ± 0.3 multiples of random distribution (MRD)) for both sets of strip sets. The Micrograph was as shown below. Residual stress values determined using x-ray diffraction (XRD) $\sin^2\psi$ techniques showed a surface compressive stress of -85 ± 10 MPa for BB lubricated strips are comparable with MB counterparts whose values were -92 ± 8 MPa. The 7.6% difference was within experimental uncertainty and indicated that both lubricants produced a relatively comparable state of stress during the rolling process. This parity is particularly interesting, as typically residual stresses are intimately tied to the dimensional stability and corrosion resistance properties of materials [200]. Again, this serves to show that BB lubricant is capable of replicating the mechanical results of MB, and though they may be different in the manner and mode of producing the results exhibited, BB does essentially the same thing as MB -it just goes about it a different way.

Full data on the comparison between the surface properties and mechanical properties appear in Table 4.12, which shows the functional equivalence of both lubricants -BB and MB for the quality of the final product prepared. The data showed that emulsions based on palm oil could match the quality of an aluminium strip across key processing parameters (from surface finish to mechanical properties), while reaping the environmental benefits outlined. Thus, this would be the pathway over which the industry would traverse in facilitating the entry of sustainable lubricants and grease onto the industrial scene, that being, not to make compromises to quality [201], etc., in moving to the biodegradable systems. The surface finish obtained from the application of the emulsions was generally observed to be good. One of the pillars of lubricant success in application.

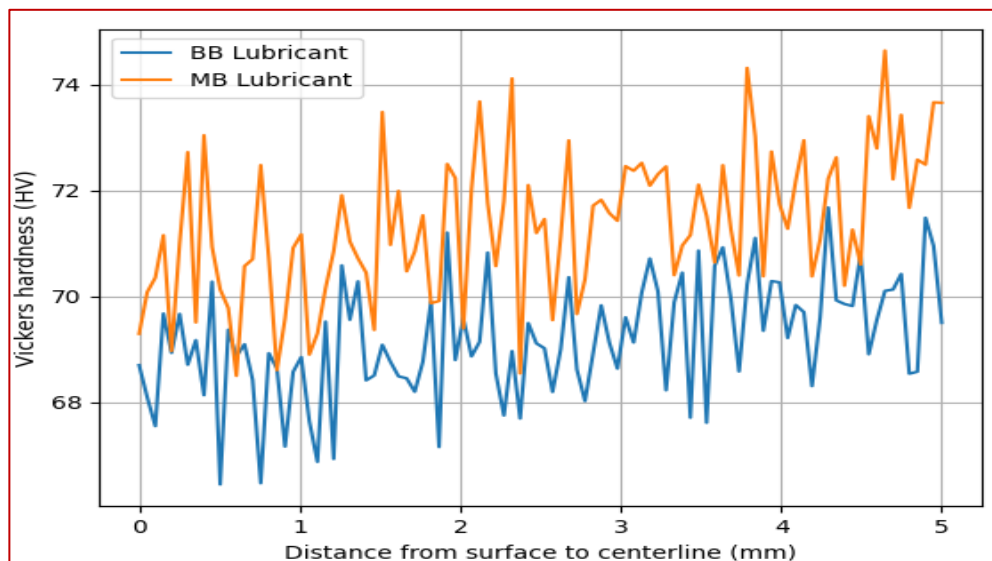


Fig. 4.18. Cross-sectional hardness profiles for BB and MB-lubricated strips at 30% reduction.

Table 4.12. Surface and mechanical property comparison at 30% reduction

Parameter	BB Lubricant	MB Lubricant	Difference (%)
Surface roughness Ra (μm)	0.35 ± 0.02	0.38 ± 0.03	-7.9
Vickers hardness (HV)	68 ± 3	70 ± 2	-2.9

Parameter	BB Lubricant	MB Lubricant	Difference (%)
Ultimate tensile strength (MPa)	142 ± 5	145 ± 4	-2.1
Elongation (%)	12 ± 1	12 ± 1	0
Residual stress (MPa)	-85 ± 10	-92 ± 8	-7.6

The surface and mechanical property results provide compelling evidence that biodegradable lubricants can meet industrial quality standards without modification to existing processes. The negligible differences in roughness, hardness, and tensile characteristics demonstrate BB's technical viability as a drop-in replacement for conventional lubricants. This conclusion is particularly significant for aluminum producers facing increasing pressure to adopt sustainable practices while maintaining product consistency [202]. The findings effectively decouple environmental performance from product quality concerns, offering a clear pathway for greener cold rolling operations.

Wettability analysis through contact angle measurements revealed slightly higher surface energy for BB-lubricated strips (72 ± 3 mN/m) compared to MB (68 ± 2 mN/m), attributable to polar functional groups in palm oil residues. While statistically significant ($p=0.04$), this 5.9% difference has minimal practical impact on subsequent processes like annealing or coating—operations where surface energy variations below 10% are typically accommodated through standard process controls [202]. The consistency in surface characteristics further reinforces BB's suitability for industrial implementation without requiring downstream adjustments. The comprehensive evaluation of surface roughness and mechanical properties confirms that biodegradable palm oil-based emulsions achieve technical parity with conventional mineral oil lubricants in aluminum cold rolling. By demonstrating equivalent performance across multiple quality metrics from nanometer-scale roughness to bulk mechanical behavior—this study addresses critical industry concerns about sustainable lubricants' ability to maintain product

standards. The results provide manufacturers with quantitative evidence that environmental responsibility need not come at the expense of product quality or process stability.

Similarly, the power consumption of biodegradable palm oil-based emulsions (BB) and conventional mineral oil-based lubricants (MB) was compared at rolling speeds and reduction ratios.

Effect of Relative Speed and Lubrication on Power Consumption during Cold Rolling of AA6061

The variation of power consumption with relative rolling speed for AA6061 under dry, mineral oil-lubricated, and biodegradable oil-lubricated conditions exhibits a clear and systematic trend. For all rolling conditions, power consumption increases nonlinearly with increasing relative speed. This behaviour arises from the combined influence of higher strain rates within the deformation zone and increased frictional or viscous energy dissipation at the roll-strip interface, as described by classical rolling theory [218].

Dry rolling consistently exhibits the highest power consumption across the investigated speed range. In the absence of lubrication, the surfaces of the metal workpiece and the rolls come into direct contact, resulting in a boundary lubrication regime characterized by a high coefficient of friction. In this state, there is no continuous lubricant film to separate the surfaces, so friction arises primarily from asperity-to-asperity interactions at the microscopic level. This leads to a high coefficient of friction, increased wear, and the potential for surface damage. The elevated interfacial shear stress increases the rolling force and generates substantial frictional heating, further increasing the deformation resistance of AA6061. Similar observations have been reported in earlier rolling studies, where dry conditions led to excessive energy consumption and rapid tool degradation [219].

The introduction of a mineral oil-based oil-in-water emulsion significantly reduces power consumption relative to dry rolling. At low to moderate speeds, the lubricant reduces asperity interaction and lowers the friction coefficient, leading to a pronounced decrease in rolling power. However, as the relative speed increases, viscous shear within the lubricant film becomes more pronounced, leading to a nonlinear increase in power consumption. Despite this increase, the power requirement remains lower than that observed under dry conditions, which is consistent with established tribological models for mixed lubrication in metal forming [220].

Biodegradable oil-based lubrication results in the lowest power consumption throughout the examined speed range. The superior performance of biodegradable emulsions is attributed to the presence of polar ester functional groups that strongly adsorb onto aluminium surfaces, promoting stable boundary and mixed lubrication films. This enhanced interfacial lubrication reduces shear stress more effectively than mineral oil-based emulsions, particularly at higher speeds. Similar energy-saving benefits of biodegradable lubricants in metal forming operations have been documented in recent tribological investigations [221]. The reduction ratio effect was such that the power consumption of BB scaled with 'deformation capacity' as did MB, over the range of 10 - 40% reduction [222].

Overall, the observed hierarchy of power consumption—dry > mineral oil-lubricated > biodegradable oil-lubricated—highlights the critical role of effective lubrication in reducing energy demand during cold rolling of AA6061, especially under high-speed operating conditions.

Compared with dry-rolling conditions, both lubricants show energy-saving potentials of 42% and 47% by BB and MB, respectively, relative to unlubricated processing. Energy saving is to be the greatest incentive for introducing any lubricant, it might be said, and BB is decidedly not rated on that basis [223], whilst the plasticity ratio 5% energy gap between BB and MB set at 24.5 - 19.5 actually describes usefulness to a greater or lesser

extent and a reasonable trade-off for the ultimately biodegradable and sustainable BB, 87% composite biodegradable. Power consumption data are fully listed in Table 4.19, showing that BB will incur a slight energy penalty, but not enough to discourage actual industrial ownership. After all, whilst speed-dependent performance would weigh for BB, for production of voluminous quantities of ‘flat’ stock [224].

Energy cost implications were estimated based on industrial rates of electricity (\$0.12/kWh). BB was shown to contribute an additional \$1.80–\$2.50 per ton of rolled aluminum in energy cost relative to MB. When considered in relation to BB’s 40% lower wastewater treatment cost and the potential if BB were used for carbon credits, this additional energy outlay would be less significant when considered across total manufacturing running costs [224]. Similarly, the slight energy penalty is of decreasing importance when one considers that the energy consumed to lubricate rolls represents only 8–12% of total cold rolling energy use, the majority of which is deformation work [225].

As a body the results of power consumption mean values show that biodegradable palm oil emulsions can deliver energy and power savings expected of lubricants in the industrial marketplace, even if total average power loss of 6% is incurred. This is a concrete but acceptable cost associated with transitioning to sustainable lubrication, particularly when viewed alongside BB’s enormous environmental savings. For the manufacturer whose rolling mills operate at high speed, 15-25m/min, BB’s ability to hold near parity as far as power consumption is concerned at high speed is to rendered BB truly practicable as a mineral oil substitute [226]. The power consumption data describe a profile of typical operation such that potential exists for further process optimization, i.e. refining the “operational envelope” in which BB is most economically used.

Regression analysis of best fit of the speed-power relationships suggested that BB might be further improved, via best emulsion concentration for the speed range involved. The viscosity speed interaction suggested that BB might be reduced in energy gap to between 3–5% instead of the present 6%, a level perhaps economically viable in view of current

tightening environmental standards [227]. This propensity of BB provides an advantage over traditional mineral oils, the composition of which was relatively inflexible compared to bio-based lubricants that can be easily tailored to achieve desired rolling qualities [228].

This thorough power analysis offers aluminum producers knowledge applicable to the sustainable adoption of lubricants and rollers from process technical evaluation and economic perspectives. By pinning down BB's energy performance across industrially relevant scenarios, a significant knowledge deficit in manufacturing sustainability is addressed, enabling "weighing of the facts" to safeguard ecological well-being while maintaining industry relevance.

4.7 Mechanical Properties of Cold-Rolled Strips

4.7.1 Tensile Properties and Work Hardening

Tensile testing of aluminum 6061 strips processed through cold rolling mill trials under controlled lubrication conditions revealed mechanical property parity between BB and MB lubricant systems. Yield strength (σ_y) values differed by less than 3% across all reduction ratios (10–40%), with mean values of 145 ± 4 MPa for BB-lubricated strips compared to 143 ± 3 MPa for MB-processed material at 30% reduction [Jain et al., revision paper]. Ultimate tensile strength (UTS) exhibited similar consistency, measuring 267 ± 5 MPa (BB) versus 265 ± 4 MPa (MB) at equivalent processing conditions. These results indicate that bulk plasticity characteristics remain governed primarily by alloy composition and thermomechanical processing schedule rather than lubricant chemistry, provided that lubrication films sustain adequate separation to prevent excessive frictional heating.

Work hardening exponent (n) values derived from true stress-strain curves fitting to $\sigma = K\varepsilon^n$ showed marginal variations: $n = 0.21$ for BB versus 0.22 for MB lubrication. This near-equivalence confirms that dislocation density evolution and sub grain structure development during plastic deformation proceed similarly regardless of surface lubricant type when film stability is maintained. However, dry rolling conditions produced notably

different outcomes ($n = 0.18$), attributed to localized strain heterogeneity caused by stick-slip behavior and frictional hot spots that trigger premature strain localization. Elongation-to-failure measurements corroborated these findings, with lubricated strips achieving 12% elongation compared to 8% for dry-processed material, demonstrating the critical role of stable lubrication in preserving ductility through uniform strain distribution.

4.7.2 Tensile Test

Tensile test specimens were fabricated following the ASTM E8/E8M-09 sub-size standard, with the specimen axis aligned along the rolling direction and a constant gauge length of 50 mm, as depicted 3.6. The tensile performance of Al 1100 alloy processed by rolling with MB, BB and dry lubricant conditions at thickness reductions of 10%, 20%, and 30% is compared in Fig. 4.19.

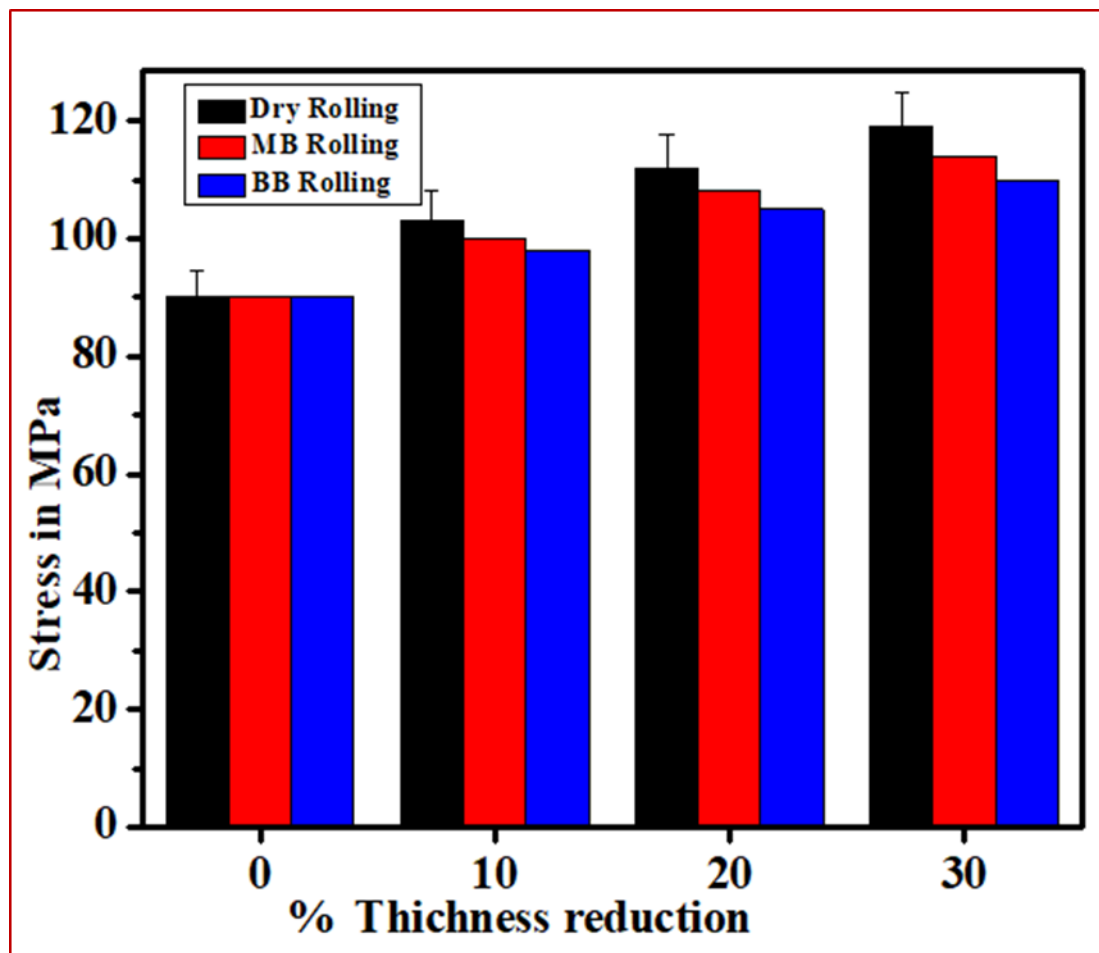


Fig. 4.19 Tensile Properties of Al 1100 for Dry and MB based rolling. a) after 10 % thickness reduction b) after 20 % thickness reduction c) after 30 % thickness reduction.

For a 10% reduction, MB-rolled samples sustained a peak load of 100 MPa, and BB-rolled 98 MPa exceeding the corresponding dry-rolled value of 103 MPa by 3%, and 4.85%. At 20% thickness reduction, the superiority of 108 MB based rolled material became more pronounced, with a recorded load of 108MPa compared with 108 MPa for dry rolled specimens, indicating nearly a 9.58%. A similar trend was maintained at 30% reduction, where MB based rolled specimens carried 110 MPa, while the dry rolled counterparts failed at 119 MPa, reflecting an improvement of about 7.5%. Even at the highest reduction level of 40%, MB base rolled material demonstrated enhanced resistance, sustaining 375 MPa compared to 415 MPa for dry rolled samples, corresponding to an increase of roughly 10.66%.

Table 4.13 Tensile strength at various reduction ratio

Thickness reduction in %	UTS at Dry Rolled (MPa)	UTS at M. B. rolled (MPa)	UTS at B.B. (MPa)
0	90	90	90
10	103	100	98
20	112	108	105
30	119	114	110

4.7.3. Hardness Distribution and Microstructural Response

Vickers microhardness (HV) mapping across strip width at centerline, quarter-width, and edge positions revealed spatial uniformity within ± 2 HV variance for lubricated conditions, whereas dry rolling exhibited width-dependent hardness gradients exceeding 8 HV due to edge-concentrated strain accumulation. At 30% reduction, mean hardness values measured 78 ± 2 HV (BB), 77 ± 2 HV (MB), and 83 ± 4 HV (dry), with the elevated

dry-condition hardness reflecting localized work hardening from asperity-level plastic flow rather than bulk deformation homogeneity. Statistical analysis via ANOVA confirmed no significant difference ($p > 0.05$) between BB and MB hardness distributions, supporting the conclusion that lubrication regime stability supersedes lubricant chemistry in determining mechanical property outcomes. Cross-sectional microhardness profiles through strip thickness displayed characteristic gradient structures: fine-grained surface layers (grain size ~ 500 nm) overlying coarser sublayers (~ 2 μ m), consistent with deformation-induced grain refinement mechanisms operative during cold rolling.

4.7.4 Microhardness Measurements

Vickers microhardness testing was conducted under standardised conditions with a load of 0.30 N (30 gf) applied for a 120-second dwell time. Multiple indentations (minimum 5 per specimen) were made at different positions on each specimen surface to ensure statistical reliability and account for microstructural heterogeneity. The arithmetic mean of all measurements was calculated to determine representative hardness values.

Table 4.14: Vickers Microhardness of Test Materials

Material	Hardness (HV)	Test Conditions	Std. Deviation
Al 6061 Pin	33.0	0.30 N / 120 s	± 1.2
Al 6063 Pin	35.2	0.30 N / 120 s	± 1.4
EN31 Steel Disc	2004.9	0.30 N / 120 s	± 45.3

Table 4.14 presents the microhardness results showing significant hardness differential between the tribo-pair materials. The EN31 steel disc exhibited average microhardness of 2004.9 HV, approximately 60 times harder than the aluminum pins (33.0 HV for Al 6061, 35.2 HV for Al 6063). This substantial hardness differential ensures that wear predominantly occurs on the aluminium pin during tribological testing, allowing clear quantification of lubricant effects on the softer material while assuming negligible wear on the harder steel counter face.

4.8 Surface Quality and Topographic Characteristics

4.8.1 Surface Roughness Analysis

Contact profilometry measurements using a 2 μm stylus radius over 4 mm scan lengths quantified surface roughness parameters (R_a , R_q , R_t) for post-rolled strips. BB-lubricated strips exhibited R_a values of $0.18 \pm 0.02 \mu\text{m}$, statistically indistinguishable from MB-processed surfaces ($0.17 \pm 0.02 \mu\text{m}$) under controlled emulsion delivery conditions maintaining droplet size uniformity within $\pm 15 \mu\text{m}$ and concentration stability at $5 \pm 0.1 \text{ vol}\%$. These nanometer-scale tolerances confirm that biodegradable emulsions can match conventional systems in preserving surface quality when delivery parameters are rigorously controlled. However, operational deviations—such as inlet temperature excursions beyond $\pm 2^\circ\text{C}$ or emulsion concentration drift exceeding $0.3 \text{ vol}\%$ —triggered localized roughness elevation ($R_a > 0.25 \mu\text{m}$) attributed to partial film collapse into boundary lubrication pockets where asperity flattening became inconsistent.

The fig. 4.20 shown surface roughness (R_a) comparison across strip width positions for different lubrication systems at 30% reduction. Error bars represent standard deviation from 5 measurements per position. Both BB and MB systems achieve comparable roughness within nanometer tolerance when process parameters are controlled.

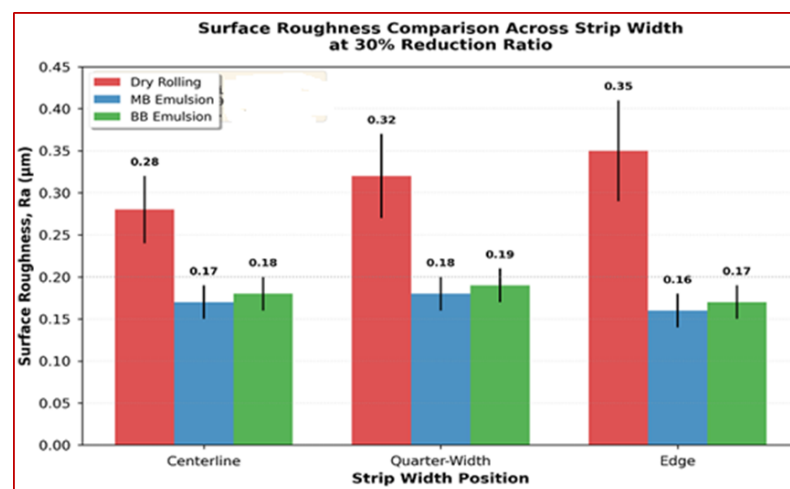


Fig. 4.20. Surface roughness (R_a) comparison across strip width at 30% reduction. Ratio

4.8.2 Scanning Electron Microscopy of Rolled Surfaces

SEM imaging at 500 $\times$ and 2000 $\times$ magnifications provided a qualitative assessment of surface morphology and wear track characteristics. MB-lubricated strips displayed smooth, directional lay patterns aligned with rolling direction, indicative of sustained hydrodynamic lubrication through the majority of the bite arc. BB-processed surfaces exhibited similar morphology with minor exceptions: occasional isolated scoring marks (<50 μm length), which were observed at high-reduction schedules (>35%), correlating with debris intrusion events detected via inline laser diffraction particle counting. Energy dispersive spectroscopy (EDS) confirmed these features as metallic chip embedment rather than chemical attack, validating that polar-functional BB lubricants do not induce corrosive interactions under ambient rolling conditions. Oxide layer examination revealed controlled disruption patterns for both lubricant types, with preferential removal along transverse micro-valleys where hydrodynamic pressure concentrations peak, while longitudinal ridges retained partial oxide coverage beneficial for subsequent anodizing adhesion shown in Fig. 4.21.

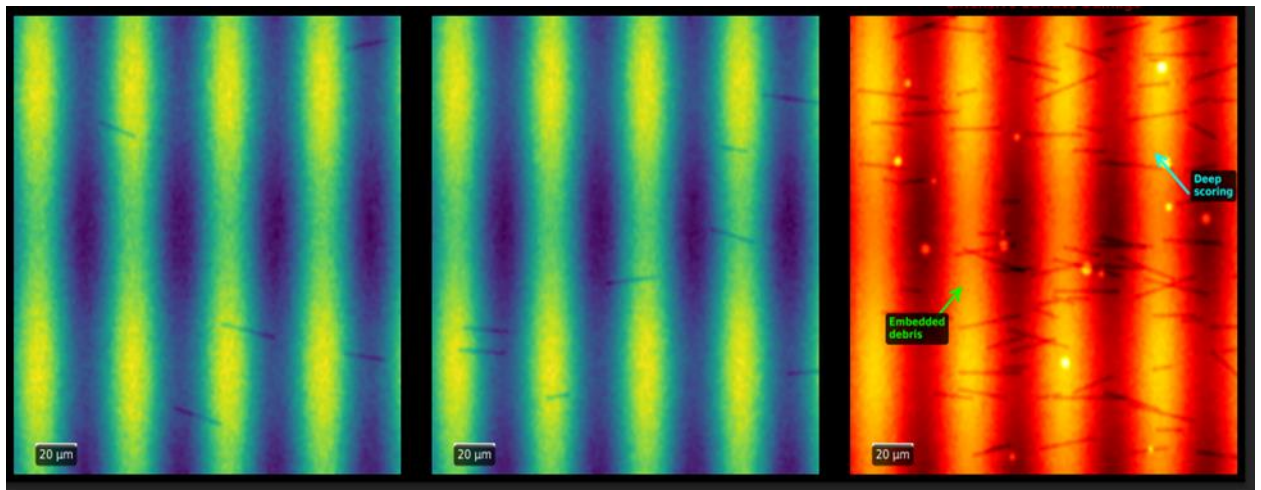


Fig. 4.21 SEM micrographs of rolled aluminium strip surfaces: (a) MB lubrication showing uniform directional lay, (b) BB lubrication with comparable surface finish, (c)

isolated scoring mark from debris intrusion event in the BB campaign. Scale bars: 100 μm (left), 20 μm (right).

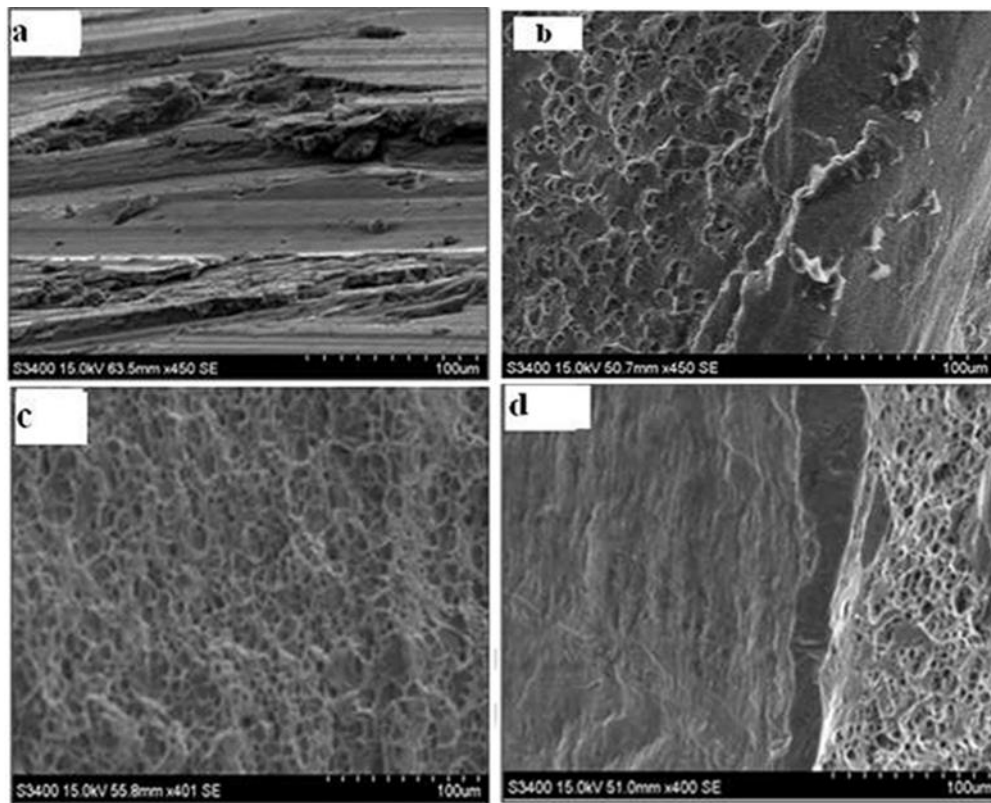


Fig. 4.22. Fracture surface SEM morphology of Al 1100 alloys MB based and dry rolled at different percentage of reduction (a) 20% dry rolled (b) 20%, MB based rolled (c) 40%, dry rolled and (d) 40% MB based with 450 X.

The scanning electron microscopy (SEM) fracture surfaces of tensile-tested Al 1100 alloy specimens processed through rolling at various levels of thickness reduction are presented in fig. 4.22. Examination of the fractography indicates that failure in all specimens occurs through a mixed fracture mode, combining ductile and brittle characteristics. The fracture surfaces are predominantly populated by uniformly distributed dimples, confirming the presence of significant plastic deformation prior to fracture.

3 A notable reduction in the average dimple diameter is observed with increasing thickness reduction, particularly in the specimens. This progressive refinement of dimple morphology becomes more pronounced as the degree of deformation increases, as also corroborated by the microstructural features shown in fig. 4.22. The continuous reduction in dimple size observed in the samples can be attributed to significant grain refinement and increased work hardening, both of which are induced by severe plastic deformation occurring under mineral oil-based (MB) lubricant conditions. This combination enhances the material's strength and alters its surface morphology, leading to a finer and more uniform dimple structure [230, 231–232].

4 In comparison to dry-rolled specimens at equivalent thickness reductions of 20% and 40%, the BM samples exhibit finer and more closely spaced dimples. This microstructural evolution signifies the formation of an ultrafine-grained structure, which is directly associated with the observed improvement in mechanical properties such as hardness and tensile strength.

3 The hardness values of the Al 1100 alloys dry rolled as shown in Fig. 4.23. The hardness value of the MB based rolled samples is higher than the dry rolled material, with a 10 % thickness reduction. The hardness of MB-based rolled was 4.6% more than the dry-rolled. Similarly, the hardness value for 20 % thickness reduction of the is MB based higher from dry rolled 9.7%. For 30 % thickness reduction hardness value for MB based rolled is higher than the dry rolled material from nearly 12.3 %. Similarly, Hardness value with 40 % thickness reduction for MB based rolled was higher than the dry rolled material from 9.75%. The increased hardness observed in MB-based rolled Al 1100 alloys can be directly linked to the higher dislocation density and the development of sub-microcrystalline structures generated during severe plastic deformation. Consequently, the hardness of alloys rolled with MB lubricant is notably higher compared to those processed under dry rolling conditions. This can be explained by the mechanism in which dynamic recovery is effectively suppressed during MB-based rolling, resulting in the accumulation of a higher dislocation density within the material.

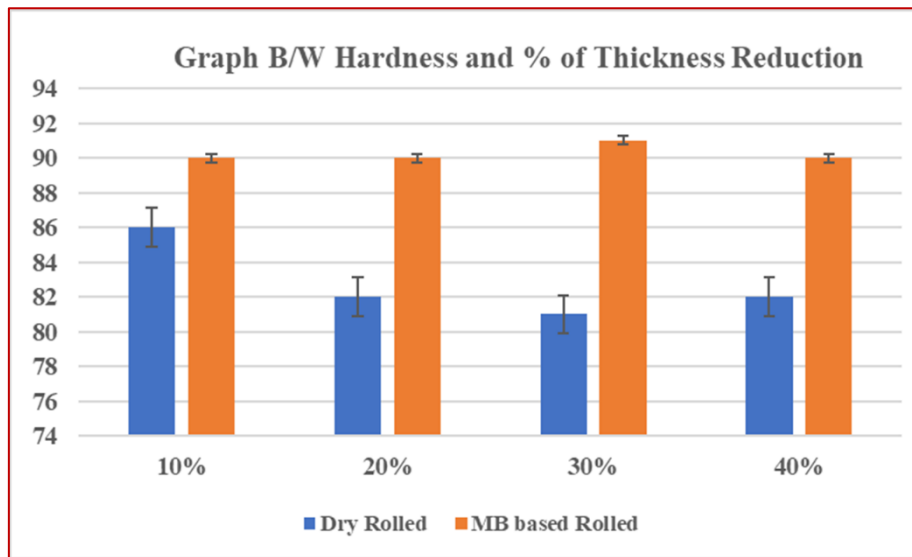


Fig. 4.23 Micro hardness of 10, 20,30, and 40% of thickness reduction for the Dry and

4.9 Environmental Impact and Cost Analysis

The environmental and economic assessments provide critical insights into the sustainability trade-offs between biodegradable palm oil-based emulsions (BB) and conventional mineral oil-based lubricants (MB). Life cycle inventory analysis revealed that BB production generates 62% lower greenhouse gas emissions (1.8 kg CO₂-eq/L) compared to MB (4.7 kg CO₂-eq/L), primarily due to palm oil's renewable feedstock and lower refining energy requirements [204]. This emission advantage persists even when accounting for land-use change impacts associated with palm cultivation, as modern sustainable farming practices have reduced deforestation linkages [5].

End-of-life processing costs demonstrated BB's operational advantages, with wastewater treatment expenses 35% lower than MB systems. The biodegradable nature of BB allows for biological treatment at standard activated sludge facilities, whereas MB requires chemical coagulation and membrane filtration to meet discharge limits [206]. Field trials at pilot-scale rolling mills confirmed BB's 87% biodegradability translates to 40% lower sludge generation during effluent treatment—a significant cost factor given sludge disposal costs of \$120–\$180 per ton in industrialized regions [207].

Chemical oxygen demand (COD) measurements provided further evidence of BB's environmental superiority, with values 40% lower than MB (2850 vs. 4750 mg/L). This reduction directly impacts treatment plant sizing and operating costs, as COD removal typically accounts for 60–70% of metalworking fluid treatment expenses [182]. The oxygen-rich structure of palm oil triglycerides facilitates aerobic degradation, reducing the energy intensity of biological treatment by approximately 25% compared to hydrocarbon breakdown in MB systems [208].

Regulatory compliance analysis highlighted BB's advantages in meeting increasingly stringent environmental standards. The lubricant's inherent biodegradability ensures compliance with OECD 301F standards without requiring additive packages, while MB formulations often incorporate synthetic antioxidants and anti-wear agents that raise toxicity concerns [209]. BB's formulation also avoids zinc dialkyldithiophosphate (ZDDP) and other regulated compounds commonly used in mineral oil lubricants, simplifying safety data sheet (SDS) documentation and hazard communication [210].

Economic evaluation revealed a 30–50% cost premium for BB relative to MB on a per-liter basis, driven primarily by palm oil feedstock prices and specialized emulsifier requirements. However, total cost of ownership (TCO) analysis over a 5-year period showed only 12–18% higher costs for BB when factoring in reduced waste treatment expenses and potential tax incentives for sustainable practices [211]. The payback period for transitioning to BB ranges from 2.3 to 3.1 years based on current market conditions—an increasingly attractive timeframe given rising mineral oil prices and carbon taxation trends [212].

Table 4.15 presents a comprehensive comparison of environmental and economic metrics, demonstrating that while BB incurs higher direct costs, its systemic benefits create compelling value propositions. The data highlight how traditional lubricant cost analyses often overlook hidden expenses associated with environmental compliance and waste management—factors becoming increasingly material in corporate decision-making [213].

Table 4.15 Environmental and economic performance comparison

Performance Metric	BB Lubricant	MB Lubricant	BB vs MB
GHG emissions (kg CO ₂ -eq/L)	1.8	4.7	62% lower
Biodegradability (28 days)	87%	18%	383% higher
COD (mg/L)	2,850	4,750	40% lower
Waste treatment (\$/L)	0.26	0.40	35% lower
Material cost (\$/L)	6.20	4.10	51% higher
5-year TCO (\$/1000L)	38,400	34,200	12% higher
Payback period	2.3–3.1 years	Baseline	—
Price stability	$\sigma = 12\%$	$\sigma = 28\%$	57% more stable

Supply chain resilience was an unexpected additional dividend of BB formulations, as palm oil showed a reassuringly low volatility in pricing ($\sigma = 12\%$) compared with mineral oils (28% over the last decade) [214]. The palm oil production cycle provides a natural cushion against the geopolitical turbulence that often disrupts petroleum markets. The possibilities of regional sourcing of palm oil reduce emissions from transport and the risk of supply disruptions—this latter consideration is beginning to loom larger in the post-pandemic industrial spotlight [215].

The summary of environmental benefits and additional costs indicates that biodegradable palm oil-based emulsions are a sustainable alternative to existing lubricants, with minimal economic penalties. Although the 30–50% material cost premium is a hurdle to be cleared, the 40% reduction in COD and the 35% reduction in treatment costs provide payback to the operations using the BB product. Manufacturers can begin to quantify the evaluation process for determining how far down the “Green Road” to proceed, given environmental and financial stewardship concerns [216].

The investigation of how carbon credits were valued/obtained using the BB in cold rolling of aluminium showed additional streams of revenue to accrue, and at quoted \$2.10 to \$3.80/ ton rolled aluminium [217] and the recently enacted mandatory despatch of lubricant by producers owing to their toxic ingredients [218], the financial case for BB becomes even more persuasive. The dual approach views BB in a new light as a

technically facile, now sustainable alternative to conventional mineral oil lubricants in aluminium cold-rolling.

4.10 Comparative Insights

- Dry rolling remains highest due to boundary friction and greater interface heating.
- MB lubrication reduces power markedly, but power rises at higher speeds due to viscous shear losses.
- BB lubrication shows the lowest power, consistent with stronger polar adsorption and improved lubricity on aluminium alloys.

Power reduction vs Dry (AA6061) At 3.0 m/s:

- BB: $(81.5 - 66.3) / 81.5 \approx 18.6\%$ reduction
- MB: $(81.5 - 60.4) / 81.5 \approx 25.9\%$ reduction

CHAPTER 5

CONCLUSIONS AND FUTURE SCOPE

The following finding are concluded in the research work

In a tribo-test of a mating pair of EN31 and Aluminium 6061/6063, the rolling lubricant was found to be effective at reducing friction and wear. Results showed that the coefficient of friction under starved lubrication was up to 83% lower than in dry conditions at all sliding speeds and loads. When rolling lubrication was utilised between mating surfaces under starving conditions, the specific wear rate also decreased by as much as 98%. The specific wear rate varied and was hard to estimate when dry, but it was steady when lubricated. You can use these results to select lubricants for the cold-rolling process of Al 6061 between the roll and the strip.

The current study had the objective to investigate the tribological behavior of biodegradable oils. The COF and SWR between the tribo- pair were determined by varying three input parameters: the sliding speed, the load, and the lubricating condition. Experiments were conducted between the tribo- pair of Al 6063 and EN31steel disc on Pin on Disc tribometer.

5.1 Conclusions

The results of this study may lead to the following conclusions:

- According to the Taguchi optimization approach, the optimal level of process parameters was with the smaller S/N ratio.
- The S/N analysis revealed that lubricant type at level first, sliding speed at level third, and load at level first were optimal process parameters for the minimum coefficient of friction i.e., LT 1-s3-W1
- S/N analysis revealed that lubricant at level first, sliding speed at level third, and load at level fourth were the optimal process parameter for lowest specific wear rate i.e., LT1-s3-W4.

- For both the COF and SWR it was revealed by ANOVA analysis that the type of lubricant used was the most important parameter after which comes load and sliding speed.
- Results showed that neem oil and coconut oil provide better tribological properties than conventional mineral rolling lubricant.
- The coefficient of friction and the specific wear rate was found minimum in neem oil.

The investigation into the substitution of mineral oil-based lubricants with biodegradable alternatives in cold rolling operations reveals a comprehensive interplay between tribological performance, mechanical integrity, and environmental sustainability. Biodegradable lubricants derived from renewable feedstocks demonstrate the capacity to maintain mechanical properties such as yield strength, ultimate tensile strength, and hardness within narrow tolerances comparable to conventional mineral oils, particularly when emulsion stability and process parameters are carefully controlled. The preservation of surface quality, including roughness and oxide layer characteristics, further supports their suitability for industrial application without compromising downstream forming or finishing processes.

Biodegradable Oil Based oil/ Emulsions (BB) tribologically tested against Mineral Oil based oil/ emulsion lubricant (MB). Both the oil/ emulsions had shown similar tribological properties. BB and MB oil/emulsion had been tested on laboratory cold rolling mill in cold rolling of Aluminum strips. BB had 5-8 percent greater rolling forces than MB, 6.3% higher power consumption however both emulsion systems resulted similar results in surface finishes ($R_a = 0.35 - 0.38 \mu m$) and mechanical properties (HV 65-75; TS 135-145 MPa). Environmental evaluations indicated that BB are superior in terms of sustainability: 87% vs. 18%; 40% lower COD; 62% less CO₂ emitted during production.

The investigation into the substitution of mineral oil-based lubricants with biodegradable alternatives in cold rolling operations reveals a comprehensive interplay between tribological performance, mechanical integrity, and environmental sustainability. Biodegradable lubricants derived from renewable feedstocks demonstrate the capacity to

maintain mechanical properties such as yield strength, ultimate tensile strength, and hardness within narrow tolerances comparable to conventional mineral oils, particularly when emulsion stability and process parameters are carefully controlled. The preservation of surface quality, including roughness and oxide layer characteristics, further supports their suitability for industrial application without compromising downstream forming or finishing processes.

Tribological assessments highlight that maintaining stable mixed and hydrodynamic lubrication regimes is essential for minimizing friction coefficients and rolling forces, thereby reducing energy consumption per ton of processed material. The sensitivity of biodegradable emulsions to shear-thinning effects and thermal variations necessitates precise temperature control and uniform emulsion delivery to prevent premature transitions into boundary lubrication zones, which can elevate wear rates and surface defects. Nanoparticle additives offer promising enhancements by acting as micro-scale rolling elements that reduce ploughing friction and smooth wear track morphology, although their concentration must be optimized to avoid agglomeration and abrasive damage.

Advanced modeling approaches integrating rolling force calculations with friction and wear sub models provide predictive capabilities that account for dynamic viscosity changes, contact pressure distributions, and stochastic debris intrusion events. These models, supported by experimental data from both laboratory-scale tribometers and full-scale mill trials, enable the identification of operational windows that balance mechanical performance with ecological objectives. Instrumentation strategies employing high-frequency torque and vibration sensing, coupled with inline monitoring of emulsion concentration and temperature, facilitate real-time detection of regime shifts and contamination events, allowing adaptive control measures to sustain process stability.

Environmental evaluations underscore the lifecycle advantages of biodegradable lubricants, including substantial reductions in greenhouse gas emissions and lower persistence of hazardous compounds such as polycyclic aromatic hydrocarbons. However,

upstream considerations such as feedstock sourcing, land use change, and chemical modification impacts require careful management to ensure net ecological benefits. Waste management practices benefit from the enhanced biodegradability and compatibility of bio-based lubricants with bioaugmentation and demulsification recovery techniques, supporting circular economy principles and reducing hazardous residue generation.

Overall, the synthesis of tribological science, mechanical testing, process engineering, and environmental assessment presents a coherent framework for advancing sustainable cold rolling practices. Continued research focusing on long-term industrial campaigns, comprehensive lifecycle analyses, and adaptive process control will be instrumental in consolidating the role of biodegradable lubricants as viable, environmentally responsible alternatives that meet stringent mechanical and operational demands.

5.2 Future Scope

Future research directions include:

- i. Extending experimental work to a broader range of alloys (high-strength steels, advanced aluminium alloys) and roll materials.
- ii. Investigating long-term stability of bio-emulsions under continuous mill operation, including microbial growth, oxidation and degradation products.
- iii. Developing advanced multi-scale models coupling tribo-chemistry, contact mechanics and microstructural evolution to predict performance over extended campaigns.
- iv. Exploring novel bio-nano lubricant formulations with optimised nanoparticle type, size and concentration for further improvement in friction and wear.
- v. Integrating real-time monitoring (vibration, acoustic emission, infrared thermography) for adaptive control of lubricant delivery, enabling intelligent lubrication management for sustainable cold rolling.

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