# **TEXTILE FINISHING THROUGH FUNCTIONALISED POLYMERS**

A Major Project Submitted to Faculty of Technology, University of Delhi Towards the Partial Fulfillment of the Requirement for Award of the Degree of

# MASTER OF ENGINEERING In POLYMER TECHNOLOGY

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Under the Kind Guidance of

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## CERTIFICATE

This is to certify that the Major Project entitled "Textile Finishing Through Functionalised Polymers" is being submitted by Mr. Rajesh Kumar Chaudhary in partial fulfillment of the requirement for the award of Master of Engineering degree in Polymer Technology to Delhi College of Engineering, University of Delhi, Delhi, is a record of bona-fide work carried out by him under our guidance and supervision.

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Place: Delhi Date:

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## **INTRODUCTION**

Consumer appeal in the textile industry is driven by functionality, comfort and visual aesthetics. The finishing stage of fabric processing therefore assumes significant importance in differential performance of a final product. A variety of polymers have been used over the years, but silicones and nitrogen–containing systems have gained prominence over the part two decades.

Textile spinning and textile weaving go back to the earliest days of India's civilization spinning and weaving are the most important industrial activities even today. The field of Indian textiles is very extensive. It may be broadly divided into plain fabrics and patterned fabrics. A variety of plain fabric were woven ranging from thick fabric to finest tissues like muslin, representing the highest achievement of cotton hand spinning and weaving industry in India. No other country ahs been able to excel the greatest beauty of these extremely fine textile. It is worth mentioning here that in spite of so much technical advancement to day, no modern textile machinery is capable of producing such fine cotton yarn. But initiated research and development activities for value addition to the textile products, for improvement in aesthetics and imparting certain desirable functional properties will must helps in future.

A vast number of synthetic emulsions are used today in the area of textile processing, including not just woven fabrics but non-woven systems and expert as well. A number of these emulsions are polymeric in nature and find application mainly in the coloration and finishing stages as printing binders or film forming surface modifiers, respectively. In this report, I have examined specific aspects of textile finishing using emulsion polymers, making an endeavor to relate structural characteristics with functional properties of the final fabric, carpet or non-woven structure, as the case may be.

## Chapter 1

## FINISHING

#### **1.1 INTRODUCTION**

In earlier times finishing of cloth (mostly cotton) meant loading it with filters e.g. starches, dextrins, china clay, French chalk whiteners, bluing agent etc.<sup>1</sup> A mixture of several such ingredients was first cooked with water to obtain a thick paste. Thus paste was then diluted to obtain desired consistency and applied on the fabric by a process known as back filling, using mangles of the stenter, dried and then subjected to calendaring. This means passing the fabric over smooth heavy, hollow, steel rollers, which are heated by high pressure steam. These processes are no longer carried out in a modern chemical process house.

The term finishing in a broad sense includes all the treatments / processes that the fabric material undergoes after leaving the loom/ knitting machine to the stage at which it enters the market. Since all fabrics produced from natural, manmade or synthetic fibers or blends thereof exhibit certain shortcomings such as lack of crease recovery, build up of static charge, flammability etc. a finishing step is normally required to enhance aesthetic properties like appearance and handle as well as functional properties e.g. dimensional stability, flame retardency etc. Thus, the principal aim of finishing is to render a fabric better equipped for a specific end use. The wear performance or serviceability of fabric depends on the following factors:

- The nature of mechanical forces encountered during use. such as bending, stretching, flexing, etc.
- Types of physico-chemical influences encountered during use, for instance exposure to intense heat, exposure to irradiation, dry-cleaning, laundering and so on
- Properties of the component fibers/yarn, fabric geometry construction, wt/meter etc.
- Chemical and surface treatments consciously imparted to or otherwise experienced by the material during processing.

The primary aim of apparel fabric finishing is improvement of the hand/feel, drapability characteristics & above all comfort properties. The performance of the finished garment during wear largely depends upon the nature of the finishing agent.

- > An ideal finishing agent should possess the following properties:
- Ability to form a suitably elastic film with adequate strength & adhesion to the substrate
- Low sensitivity to changes in relative humidity.

Not be easily removable through washing, dry cleaning or mechanical abrasion.

Clearly, it is not enough to have merely understood the important requirements of a good finishing agent unless it were possible to understand and co-relate the performance of various finishing formulations with respect to their chemical structure and composition. In this context, a key ingredient of a finishing agent is the film forming polymer, which is usually delivered to the fabric from an aqueous phase in which it is uniformly dispersed.

With changing lifestyles and consumer preferences, it has progressively become essential that textile products are designed according to end-user need. The **Kawabata Evaluation System for Fabrics (KEF-S)** is an interesting tool for engineering textile products towards fulfillment of consumer need by enabling optimisation of fiber mix, process parameters and finishing treatments.<sup>2</sup> It takes into account the fact that the handle of fabric depends mainly on its low stress mechanical properties such as resistance to shear, bending, tensile and compressive forces, surface friction etc.

The term handle or **'Hand'** refers to a subjective assessment of textile material obtained from the sense of touch. It implies the ability of fingers and of the mind to make a sensitive and discriminating assessment and to integrate and finally to express the results in a single valued judgement. The Kawabata system is based on the measurement of these properties.<sup>3</sup>

The low stress mechanical properties of the fabric in turn depend upon the fiber yarn and fabric properties as well as the conditions employed for manufacturing of component fiber / yarn. Moreover the conditions employed for manufacture of the fabric, nature and quantity as well as the method of application of the chemical also affect the handle.

For example, increase of fiber diameter results in:

- ✤ Increase of stiffness (Koshi)
- Increase of anti draping stiffness (Hari)
- ✤ Increase of crispness (Shari) and
- Decrees of fullness and softness (Furukami).

The Kawabata system also measures **Total Hand Value (THV)**, which is derived by considering Koshi.Numeri (smoothness), Furukami and two more factors namely Hari and Shari.

Before the advent of KEFS ,3 Parameters namely bending length / moment drivability and resilience are used to characteristics the finish. The bending length / moment was used as a measure of stiffness. Drapability was used as a measure of body and crease recovery angle was used as a measure of resiliency.

It is quite clear from the above discussion that the handle of the fabric cannot be easily quantified and the predictive value of the above a parameters is quite limited.

Bending length defined by **Pierce** is the cube root of flexural rigidity divided by the weight per unit area. In other words bending length of a fabric is the length, which will bend under its own weight to a definite extent.<sup>4</sup>

There are two types of methods for determination of bending length. The first type of methods is dependent on the relation ship between the bending moment per unit width of the material and the radius of curvature.

Bending moment is defined as the force required to bend a strip of ideal material and is proportional to the reciprocal of radius of curvature.

The second set of methods measures the bending stiffness without relating it to the curvature. A number of equipments e.g. Schiefer Flexometer, Gurley Stiffness Tester, Drapeometer and Pierce Hanging Heart Loop Tester have been designed over the years. The ASTM test for measurement of the stiffness of fabric and the British Standard Method are developed from Pierce Test.

Pierce states that the way in which the fabric drapes or hangs depends largely on its stiffness and on its weight. The bending length defined as above ,may be regarded as a quantitative measure of the property on which the hang depends.

Although starches of various kinds have been popularly used as finishing agents from time immemorial, mainly due to low cost and ample availability, starches are not considered suitable for a number of modern finishing requirements. A starch film is not elastic, tends to be brittle, has high sensitivity to changes in relative humidity and has low durability to washing/laundering and use of the garment.

The main components of a finishing bath are:

Stiffening agents and

- Binding agents like starch to avoid dusting out the material such as China Clay. It also includes
- Filters (clay), softening agents is and other additions for specific uses such as fire – proofing, mildew – proofing etc. the nature of the material to be finished, e.g. voiles are given no calendaring. The hard and wiry effect produced is maintained after finishing. For long clothes, softening agents are added and the cloth calendared to give a full and lustrous effect.

#### **1.2 METHODS OF FINISHING**

Finishing methods include chemical modifications of the materials and imparting to it dimensional stability, resistance and anti shrink properties. These methods have a tendency to emphasize any inherent defects present in the fabric, such as nonparallel warps, missing picks and other faults of weaving. In India the older conventional finessing processes are used to a large extent. With introduction of synthetic fibers and the various blends with natural fibers, new processes and machinery have been devised.

Finishing processes can be classified by different ways, as shown in chart .

- Mechanical / Physical methods .
- Chemical methods

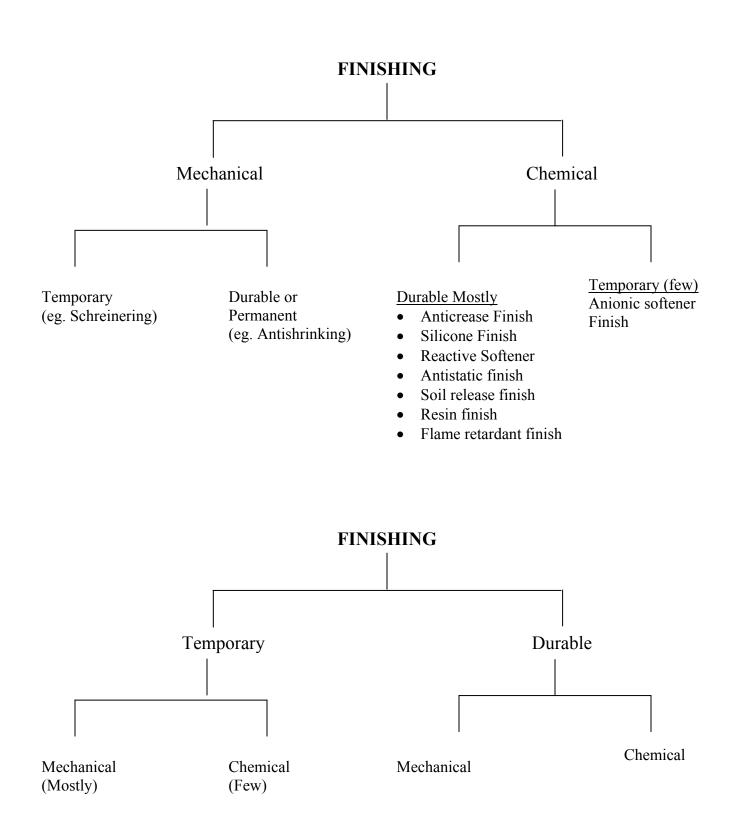
- ➤ Temporary finishes
- Semi-permanent finishes
- Permanent finishes

Physical methods may range form a simple drying operation on drying cylinders or on a stenter to a complicated series of calendaring operations (schreiner, chasing and friction). As for and lofty feel may be obtained by raising the surface of the cloth or a mellow feel by breaking the finishing of filled goods. In addition, other machines are available for controlled and compressive shrinkage.

Chemical methods involve application or deposition of chemicals or carrying out a chemical reaction on the fabric. Many commercial compounds such as starches, gums, china clay, oils etc. are applied to cotton fabrics, combined with mechanical finishes. In addition to these, long chain fatty acids, synthetic resins, cellulose derivatives, quaternary ammonium compounds, etc. are also used. The last three types of compounds produced more durable effect and are employed for imparting permanent effects. The stability of various finishes to washing various depending on the finish employed, but no finish lasts indefinitely; neither is it required nor expected to do so.

A finish is permanent, if it remains unaffected through all the conditions of wear and treatments to which the fabric may be subjected to in the normal life. The finish need not outlast the material on which it is applied. Thus, the durability of the finish depends on the life of the fabric and its specific field of use. On this basis of temporary and permanent effects, the following may be **transient or impermanent** finishes:

- Mechanical: Some Mechanical finishes are calendaring, beetling, schreinering, embossing and glazing, breaking, stretching etc.
- Filling: Filling agents such as starch, gum, Epsom salt, China Clay and other mineral fillers may be used to give body to the material.
- Softening: Softening agents such as oils, fats, waxes soaps, polyethylene, cationic softeners, deliquescent substances such as glycerine, glucose, magnesium chloride etc. may be used.



#### **1.3 INGREDIENTS AND ADDITIVES**

A number of chemicals, usually known as textile finishing chemicals, are used in various processes to which grey, bleached, dyed and / or printed fabrics are subjected, to get a variety of useful effects suiting different end-users.

These finishing chemicals may be divided as follows:

## (a) Stiffening Agents

These include thickeners like corn starch, wheat starch maize starch, potato starch (farina), tapioca, arrow root starch, sago, dextrin, soluble starch, gums, casein, gelatine, glue, polyvinyl acetate, carboxymethyl cellulose, hydroxyethyl cellulose, methyl cellulose, polyacrylamide, polyacrylates, etc.

## (a) Softeners

These include glycerine, glucose, fatty matters, tallow, paraffin, stearin, spermaccti, coconut oil, palm oil, olive oil, beesewax, japan wax, soap. Turkey Red Oil, nonionic softeners, anionic softeners, cationic softeners, reactive softeners, polyethylene emoulsion, silicone emulsion, etc.

## (b) Weighting Materials

Calcium sulphate, gypsum, magnesium sulphate, Glauber's salt, barium sulphate, lead sulphate, zinc sulphate, barium carbonate, barium chloride, zinc chloride, china clay, browh clay, talc, steatite, etc. are included in this group.

## (c) Antiseptics

These include alum, boric acid, phenol, pentachlorophenol, creosote, borax, oxalic acid, salicylic acid, zinc chloride, zinc sulphate, sodium silicofluoride, salicylanilide, magnesium silicofluoride and silicofluorides of aluminium, ammonium and antimony, dichlorobenzene, sulphon – metylamide, DDT (Dichloro Diphenyl Trichloroethane), cadmium selenide and sulphocyanide, tellurides of antimoney, bismuth, cadmium, cobalt and copper, N-(tributyl plumbyl) imidazole, tri(octadecyl)-methyl ammonium chloride, zirconyl ammonium carbonate (which produces zirconia on heating and then reacting with chromium and copper salts), dieldrin etc. These are variously known as rot-proofing, moth-proofing and mildew-proofing agents or preservatives.

### (e) Water – Proofing Agents

For the purpose, vulcanized natural rubber, oxidized oils or varnishes, polyvinyl chloroacetate, cellulose acetate, polyvinyl butyral, polyvinylidene chloride, etc. are used.

#### (f) Water – Repellants

These include metallic soaps like aluminium stearate (made form aluminium acetate and sodium stearate), paraffin wax or vegetable waxes, zirconium soap, ammonium zirconyl carbonate, chlorinated paraffin waxes, micro-crystalline waxes, methylol stearamide, silicone emulsion (along with zinc acetate catalyst)etc.

#### (g) Flame – Retardants

Also known as fire-retardants, which include water soluble compounds of phosphorus, antimony and boron, especially phosphates and borates (for temporary flame retardancy), phosphorus – containing methylol compounds like tetrakis (hydroxymethyl) phosphonium chloride and hydroxide (THPC and THPOH), aqueous dispersion of decabromobiphenyl oxide and Sb<sub>2</sub>O<sub>3</sub> with an acrylic latex binder, stable emulsions of cyanamide – phosphoric acid mixture, tries (2,3-dibromopropyl) phosphate, oligomeric vinyl phosponate (permanent flame retardants), etc.

## (h) Crease – Resisting and Cross Linking Agents

These include dimethylol urea (DMU) also called urea formaldehyde precondensate), polymethylol melamine (melamine–formaldehyde precondenstate), Dimethylol ethylene urea (DMEU), dimethylol propylene urea (DMPU), dimethylol dihydroxy ethylene urea (DMEU), dimethylol propylene urea (DMPU), dimethylol dihydroxy ethylene urea (DMDHEU), methylolalkyl triazone, etc. These are used along with catalysts chosen form diammonium hydrogen phosphate, magnesium chloride, zinc nitrate, citric acid magnesium chloride mixture, ammonium sulphamate, etc.

#### (i) Soil Release Agents

These include acrylic copolymers (e.g. 70% methacrylic acid and 30% ethyl acrylatel), fluorochemical-based chemicals (e.g. polyacrylates in which the alcohol portion contains fluorine atoms instead of hydrogen attached to carbon atoms), reaction product of tetrakis (hydroxymethyl) phosphonium chloride and

1.1-dihydroperfluorooctylamine (POA) (applied on cellulosic textiles followed by ammonia cure) etc.

## (j) Antistatic Agents

These include polyethylene glycol mono-or di-esters (with lauric, oleic, or stearic acid), salts of high molecular weight fatty amide – fatty alcohol – ethylene oxide condensates, dialkyl dimethyl ammonium chloride, (alkyl being  $C_{8-18}$  chain), stearamide of higher polyethyleneimine, polyethylene glycol ester of rosin (abietic acid-based product) etc.

## (k) Wool Shrink – Resisting Agents

These include reactive pre-polymers containing carbamoyl sulphate groups, isocyanate-terminated urethane prepolymer, dichloroisocyanuric acid (DCCA) and its sodium or potassium salts.

## (l) Fluorochemical Repellents

For imparting both oil-and water-repellency to textiles fluoropolymers such as vinyl polyemers of acrylic or methacrylic type, copolymers of vinyl esters, vinyl ethers, allyl ethers and thiomethacrylates also have been patented. Vinyl polymers consist of perfluoroalkyl group, polymer backbone and a link between the two. They are also made by copolymerizaing one or more fluorinated monomers with one or more monomers not containing fluorine. The structure of non-fluorinated monomers and their ratio to fluorinated monomer affect repellency and otter properties. Comonomers with a crosslinking function (hydrox, epoxy, vinyl) are used to increase the durability of repellent polymer.

#### (m) Antipilling Agents

For Pilling is an unpleasant phenomenon usually associated with spun yarn fabrics, especially those containing synthetic fibres. In this, fibres are released from the yarn by bending and abrasion and they combine together at the surface of the fabric forming knots, known as pills. These pills contain fibres that are still partially embedded in the yarn and hence are secured to the fabric surface. Formation of polymer film such as polyvinyl propionate, polyacrylates etc. and resin finishing reduce pilling. Synthetic resin dispersions are also effective antipilling agents.

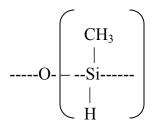
#### 1.4 SILICONES FINISHING

Silicones are broad family of synthetic polymers that are partly inorganic and partly organic<sup>5</sup>. Their structure consists of alternating silicone and oxygen atoms. Typically, one or more of the organic groups are attached to the silicon silicon atmos, imparting properties such as chemical resistance, lubricity, improved thermal and oxidative stability, and reactivity with organic materials and polymers. Silicones are grouped into three major categories: fluids, resins and elastomers(6-7). In recent years, silicones have grown from a chemical rarity into a widely used family of products (9).

They have proved extremely versatile in solving many industrial problems and have been key ingredients in a number of new products. No other synthetic fluid has found such a variety of uses in so many different industries (10).

Silicones have broad application in textile processing and finishing (5,11-12). Most of the products of this industry are based on polydimenthylsiloxane (PDMS) and their organic modifications. The applications of silicones vary widely and include fabric finishes, coatings, defamers, print paste softeners, etc.

Two most important polysiloxanes having applications in the textile industry (softening and water repellency) are methyl hydrogen and dimethyl derivatives.



Methyl hydrogen derivative



These derivatives can undergo further polymerizations chain extension and self cross linking reactions. In the textile applications both the latter properties are considered undesirable and these can be prevented by replacing the hydrogen atoms by more inert groups such as methyl groups. Then such polymers are said to end blocked. Thus, two types of end-blocked polysiloxanes are trimethyl siloxy end blocked dimethyl polysiloxane or linear dimethyl polysiloxane, i.e.,

$$O \quad \begin{pmatrix} CH_3 \\ | \\ Si - O \\ | \\ CH_3 \end{pmatrix} = \begin{pmatrix} CH_3 \\ | \\ Si - CH_3 \\ n \\ CH_3 \end{pmatrix}$$

and trimethly siloxy end blocked methyl hydrogen polysiloxane i.e.

$$O \left( \begin{array}{c} H \\ | \\ -Si--O \\ | \\ CH_3 \end{array} \right) \xrightarrow{\text{CH}_3} Si-CH_3 \\ | \\ Si-CH_3 \\ | \\ CH_3 \end{array}$$

The polymer of dimethyl polysiloxane is formed at a temperature of  $200-250^{\circ}$ C, but this is rather too high and is not suitable for textile applications. The product trimethyl siloxy end blocked methyl hydrogen polysiloxane can be cured at about  $160^{\circ}$ C, but it has an adverse effect on fabric hand and, therefore, the mixture of two is used.

A commercial silicone product consists of about 60% by weight of trimethyl siloxy end blocked methyl hydrogen polysiloxane (DP, 500-600) and 40% by weight of trimethyl siloxy end blocked dimethyl polysiloxane (DP 40-60) having a viscosity of about 12,500 cPs at 25<sup>o</sup>C. While the former contributes towards durability by reaction with the fibre substance the latter imparts luibricity and hydrophobicity. Metallic salts such as zinc acetate, butyl titanate or organoamines are used as a catalyst for effecting the polymerization. These

are cured by a condensation reaction in which oxygen bridges are formed between the chains by the removal of water. A strong bond is created between the polysiloxane and the fibre surface which probably arises from chemical combination between the polysiloxane and hydroxy or amino groups present in the fibre molecule very near to the fibre surface, or between a very thin film of moisture which is in very close association with it.

#### **1.5 BIO FINISHING / ENZYME FINISHING**

Enzymes are complex proteins secreted by living organisms. These are naturally occurring high molecular weight proteins capable of catalyzing specific chemical reaction. They are also known as biocatalysts.

## **General Mechanism of Enzyme Activity**

The enzyme catalyzes the reaction at the substrate surface. Enzymes contain true activity centers in the form known as fissures, holes, pockets or cavities. The active site in the part of the enzyme molecule that combines with substrate. The active site of the enzyme recognizes the corresponding site of the substrate molecule. This makes the enzyme specific towards substrate and allows proper orientation of both the molecules so that the active site of the enzyme molecule has access to the appropriate site of substrate molecule. Thus enzyme form an enzyme substrate complex.. This is known as 'lock and key' mechanism.

## **Enzymes for Textile Application**

Some of the enzyme classes, which are suitable for textile applications, are as follows:

Process	Enzymes
Desizing	Amylase
Scouring	Pectinase, lipase
Bleaching	Oxidoreductase, Xylanase
Dyeing	Oxidoreductase
Finessing	Cellulase
Wool scouring, silk degumming	Protein age

## **Finishing of Cotton**

Cellulase is the most widely used enzyme in the finishing of cellulosic fiber. This enzyme is used in bio-polishing of cellulosic fabrics to remove surface fibres and to promote softness. Cellulase is also used in laundering of indigo dyed denim garments to produce the worm look that is held in such a high regard by the consumer. During this process of stone less stone washing the cellulase catalyses the hydrolysis of fiber surface, releasing indigo from the fabric in an uneven manner.

Since cellulase results in weakening of the fiber. An enzymatic system based on oxidoreductase has been developed. In this system, the enzyme catalyses the oxidative destruction of indigo, without harming cotton fiber.

One of the novel uses of enzymes is cross-linking of cotton. It is well known at lipases are used to catalyse the hydrolysis of fats, these enzymes can be made to work in reverse when no water is present. It is possible that suitable lipases or other enzymes can be found that will catalyze cross-linking reactions between polybasic organic acids or other cross-linking agents and cellulose to produce durable press fabrics.

## 1.6 C-H-C PROCESS

This process improves both visual and tactile properties of fabrics made of Dacron polyester fibre (Type 54: Staple and Type 56: Continuous Filament) as claimed by DuPont. Commonly called the C-H-C Process it improves the liveliness, drape and covering power. In addition it gives the fabric a drier and warmer hand than that of an untreated fabric. The process was originally developed for use on 100% Dacron fabrics. Subsequent work, however, proved it to be equally beneficial when applied to blend fabrics of Dacron (exclusive of blends with protein fibres).

In the C-H-C process the fabric is calendared heat set and then treated in a solution of caustic soda. The fabric after usual scouring is calendared at 90- $175^{0}$ C under 10-20 tonnes pressure at a speed of 15-\*20 m/min., with one pass on each side calendaring flattens the yarn bundles and consequently increases the covering power of the fabric. It also reduces the thickness of the fabric making it light and airy. Heat – setting is then carried out under standard

condition and using hot roll, radiant heat or hot – air equipment. This is followed by caustic soda treatment which removes plastic like film form the fabric surface and improves and liveliness and drape. Actually, caustic soda dissolves the fiber surface and is accompanied by loss in weight. The degree of improvement in fabric properties closely related to the weight-loss obtained during caustic treatment.

The fabric is entered at room temperature in a three percent caustic soda solution (on the weight of the bath). The temperature is raised to 90-100°C and the treatment continued at this temperature for 30 minutes. The bath is cooled to 70°C over a period of 20 minutes and then dropped. The fabric is washed in cold water for 5 minutes, rinsed for 5 minutes a bath containing 2 gm/liter glacial acetic acid, and then finally washed with cold water and dried.

#### **1.7 FLAME RETARDANT FINISHES**

Flame retardant finishes can be defined as the finishes which appreciably slow down the combustion of a treated material in comparison to an untreated one, once the source of heat is removed.

Combustion of any material is essentially a vapour phase oxidation. Actually the fabric which is a solid material undergoes several stages prior to the oxidation or combustion and these may be distinguished as heating, decomposition, evaporation and oxidation. The possibility of making fabric flame retardant exists at all these stages. Persistent trends in flammability regulations have brought tremendous pressures on textile and chemical industries to develop fibres and finishes which can withstand the requirements of these legislations. A fabric can be rendered flame retardant in the following three ways:

- Fibre can be so chosen that it is inherently flame retardant, e.g., Nomox (nylon) fibre of DuPont.
- 2. Flame retardants can be alloyed with the fibres.
- 3. Flame retardants finish can be applied to the fabric. It may be durable or otherwise.

Whenever a fabric is made from an inherent non-flammable fibre, the durability of the flame retardancy is not a problem. Durable flame retardant finishes have been developed for cellulosic fibres. The advantage with these fibres is that they contain groups which are capable of reacting with acids released by solid phase flame retardants and these materials, therefore, can be made flame retardant with the compounds containing bromine (Br), phosphorus (P), sulphur (S), or boron (B). Synthetic fibres such as polyester either do not contain reactive sites or fibres such as polyester either do not containing Br, P, S or B. Therefore, the most effective flame retardant compounds for polyester would be those which suppress combustion at the oxidation stage, i.e., vapour phase flame retardants.

The other approach for making polyester flame retardant could be the addition of flame retardant compounds either during polymerization or to polyester melt before spinning. Flame retardant can be applied to polyester by thermosoling or by exhaust process.

The problem of rendering-polyester/cellulosic blends flame retardant is still more severe because of the fact that the charred cellulosies serve as a framework for melting polyester. Therefore, the problem of rendering polyester/ cellulosic blends flame retardant covers two complex areas, viz., selection of a flame retardant system which is capable of imparting flame retardancy on both polyester and cellulosics and method of application of this system.

## **Requirements of Flame Retardant (FR) Finishes**

A flame retardant finish must have the following characteristics :

- Heat and hydrolytic stability
- ✤ High water insolubility
- ✤ Should not impart any colour to the finished fabric.
- Should not have adverse effect on 'hand' of the fabric.
- ✤ Capability of attaching to cellulosic and synthetics.
- ✤ Non-toxic, and
- Should be applicable by conventional machinery

However, it is not possible to have all these properties in single compound and, therefore, a compromise to achieve the best should be made.

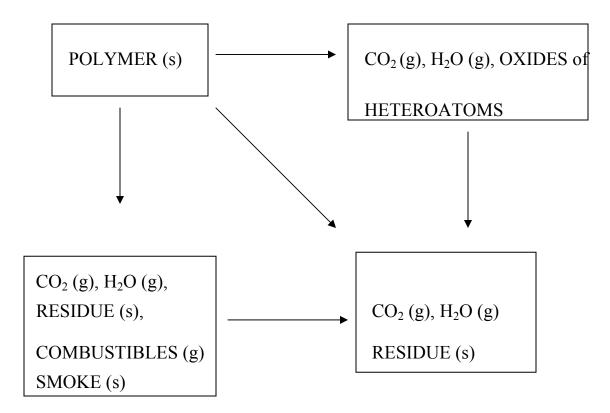
## **Mechanism of FR Finishes**

Two types of flame retardant finishes are available- solid phase and vapour phase,

*Solid phase Finishes* : These are based on phosphorus sulphur and boron. These compounds, on heating are decomposed into sulphuric acid, phosphoric acid and boric acid respectively. It is these acids which react with the hydroxyl group of cellulosics to form esters which are non-volatile in nature and, therefore, suppress the combustion of the material.

*Vapour Phase Finishes :* These compounds are halogenated materials and are used on synthetics which on heating decompose and give free radicals. These free radicals combine with the oxygen of the atmosphere. The reaction is complex in nature. The oxygen thus combined is not available for oxidation of flammable gases generated by the decomposition and evaporation of the substrate and hence the combustion is suppressed.

Calorimetric Combustion Scheme<sup>30</sup> for the two types of polymers is shown below. In case of solid phase finishes  $(\Delta H_2 / (\Delta H_C^0)_P)$  decreases with increasing concentration of polymer, whereas in case of vapour phase finishes ( $\Delta H_1 / (\Delta H_C^0)_P$ ) decreases with increasing concentration of polymer.



$$(\Delta H_{\rm C}^{\rm O})_{\rm P} = \Delta H_2 + R \ (\Delta H_{\rm C}^{\rm O})_{\rm R}$$

 $\Delta H_2 == \Delta H_1 + \Delta H_3$ 

H<sub>1</sub>= Actul Heat Released in Air-Callg Polymer

H<sub>2</sub>= total Heat Fuel Generated-Callg Polymer

H<sub>3</sub>= Heat and Combustion and Combustibles- Callg polymer

 $(\Delta H^{o}_{c})p$  = Heat of Combustion of Polymer-Callg Polymer

 $(\Delta H^{o}_{c})_{R}$  = Heat of Combustion of Residue- Callg Polymer

 $H_2 = (\Delta H^o_c)_p - RX (\Delta H^o_c)_R$ 

Fig. : Calorimetric Combustion Scheme for Polymers

## **1.8 SOIL RELEASE FINISHES**

The soiling of textiles is one of the most difficult problems associated with their use. Cotton garments and for that matter cellulosics as well, do not pose problem of soiling because of their high moisture regain. Nevertheless the problem is encountered with resin finished cellulosic fabrics. But fabrics rich in synthetics show tendency towards soiling and pose problems during their use.

The soiling of fabrics is due to:

- (a) Interfacial attraction or Van-der-Waal forces,
- (b) Electrostatic attraction,
- (c) Mechanical forces and
- (d) Hydrophobicity or Oleophilicity.

There are two of soily matters- dry or particulate soil and oily or greasy soil. The former which includes particles of dust, sand, earth, soot. metallic oxides and carbon with tarry substances may be hydrophilic (metallic oxide) or hydrophobic (carbon) in nature. The latter includes glycerides, long chain fatty acids and alcohols, lubricating oil, etc. which are mostly hydrophobic.

## **Soiling Mechanism**

Fabric soiling takes place through three different mechanisms:

- ➢ Dry soiling,
- ➢ Spot soiling and

## ➢ Wet soil redeposition.

Most of the commonly used man made fibres for apparel purposes have low moisture regain as a result of which they accumulate static charges. This leads to the attraction of dust and dirt particles from the air leading to **dry soiling**.

**Spot soiling** is localized stain on the fabric and can be due to a variety of substances capable of penetrating into the sub-microscopic interstices of the fibre. The extent of soiling depends upon variables such as surface activity of the soil, viscosity of the soil, etc., Two major factors which influence the degree of soiling are the time of contact between the soil and the fabric and the amount of pressure applied to the soil while in contact with the fabric.

Soiling due to **wet soil redeposition** from wash water is the cause of commonly occurring graying of fabrics. In polyester-cellulosic blends, this problem can be aggravated by a number of easy finishing agents, which are used to impart permanent press properties to the fabrics.

Factors Affecting Soiling:

- (a) Physical factors
- (b) Choice of washing medium
- (c) Fibre type
- (d) Finishes of the fabric

## **Commercial SR Finshes**

Carboxymethyl cellulose (CMC) is known for its soil release properties for many years. Addition of CMC in rinse water makes the soil release easier. Charge on the surface and hydrophilic nature of CMC are important factors contributing to the ease of soil removal. CMC because of its high molecular weight remains mostly on the surface of the fibres. Extensive work has been done in this field and in the last 10 years many SR finishes have appeared all over the world. These SR finishes are generally classified into following three classes :

- ✤ Acrylic polymer finishes
- Fluorochemical finishes
- ✤ Oxyethylene products of polymers

## **Acrylic SR Finishes**

Soil release finishes based on mixed polymers of acrylic acid, substituted acrylic esters and methylolated acrylamide have been marketed under the trade names of Perapret D (BASF), Rhoplex 528 and Rhoplex SR 488 (Rohn and Hass). The durability of acrylic SR finish can be improved by incorporating dimethylolcihydroxy ethylene urea (DMDHEU) in the bath. Acrylic SR finishes are hydrophilic and anionic in nature. The hydrophilicity of the finish helps in reducing the affinity of the fibre for hydrophobic soils. The anionic nature of the finish in alkaline wash solutions helps in repelling the negatively charged particulate soil from the fabric. These finishes are used on light and medium weight fabrics.

#### **1.9 ANTI-PILLING TREATMENT**

Pilling denotes the formation of bundles of entangled fibres on fabric surfaces as a result of continuous mild rubbing or abrasive action during its wear and washing. The formation of pills is caused by the fibres which naturally protrude from the surface of the fabric or by those which migrate out of the spun yarns on the surface of the garments. These fibres then entangle to form pills. Alternatively, pill formation can occur in case of very long fibres which would tease out from yarn structure and form an entangled roil.

On further abrasion the pills may or may not detach from the garment depending upon the resultant net effect of two opposing processes, namely, pill formation and wearing off of the pills. If the external force is greater than the strength and abrasion resistance, etc. of the fibres, pills would wear off and vice versa. The relative magnitude of these two forces may vary with time in a given fabric and also vary from fabric to fabric depending upon the properties of the constituent fibres. Therefore, the garments made from stronger man-made fibres such as polyester or polyesterblended with cellulosic fibres, the pills are tenaciously anchored to the surface and hardly rub off during wear. But in case of fabric made of comparatively weaker fibres such as cotton and viscose, the pills wear off as a result of destructive abrasion and no pilling is visible on the surface.

As there is a difference among fibres in their net rates of pill formation, there is an obvious difference in the character of pills formed. The pills of 100% wool, spun viscose or cotton contain mechanically damaged fibres and with relatively few anchoring fibres. The strong synthetic fibres, on the other hand, generally form more compact and tighter pills and these contain portions of all types of fibres present and are held on surface by strong anchoring fibres. The hydrophobic fibres, because of their electrostatic properties, attract lint or foreign matter, which can act as a pill nucleus and thus greatly aggravate the problem.

Pills rarely affect the actual durability of the garments but they do spoil the appearance of a garment and render expensive garments quite useless. Pilling is a severe problem with the fabrics made of synthetic fibres, their blends and lately with carbonized polyester fabrics. A large amount of work has been done in several technological laboratories and also in the textile industry to determine the factors particularly responsible for pilling and to develop practical and effective measures to obviate the onset of pill formation.

## Chapter 2

# **MODERN SYNTHETIC FINISHES**

## 2.1 INTRODUCTION

Modern synthetic finishing agents fall into three broad categories:

- I. Surfactant based softeners, which may be cationic / nonionic/ amphoteric in nature,
- II. Polyalkyl siloxanes, aminosilicones and other silicone derivatives, and
- III. Film forming emulsion polymers, with or without incorporation of plasticisers.

Here, I will confine my attention to last-mentioned polymeric systems:

- For instance, printed PET sarees made form highly twisted filament yarn are subecited to finishing with a combination of polymeric stiffening agent and body building agent to endow them with an almost traditional, cotton like comfort and feel.
- Economy suiting material (wt per meter ~200-250 gm) are subjected to finishing with a view to impart fullness of body, better draping properties and richness of texture. In such applications several ingredients e.g. stiffener, body builders, smoothening agents and

softeners are included to produce the desired functional and aesthetic properties.

Interlinings, which can be made form both woven and non-woven material, are used extensively in garments with a view to enhance aesthetic appeal and comfort besides providing mechanical support to the shell fabric. Cuffs and collars normally contain such inserts. These come in a variety of stiff, soft and very soft handle, delivered as required by use of suitably designed polymeric binders.

The ultimate properties imparted to a textile (fabric or non-woven structure) by a polymer are dependent on:

- Primary composition of polymer chain (molecular weight distribution, monomer distribution and ragiochemistry)
- Presence of covalent cross links between the polymer and the fiber.

Similarly physical bonds between the polymer and fiber including hydrogen bonds, London Forces and other attractive forces also have some influence on the ultimate web properties, notably stiffness and drape characteristics.

Various investigators have developed correlations between the handle and tensile properties of the film and as well as with the glass transition temperature  $(T_g)$  of the deposited polymer.<sup>13-15</sup> The theoretical  $T_g$  can be calculated form the monomer composition while the actual  $T_g$  is measured by various phase change detection technique; there is good correlation between measured and calculated

values. An issue arises here wherein, although film properties are well correlated with polymer  $T_g$ , the handle of the web and other fabric properties cannot be adequately extrapolated from this correlation.

There are a number of reasons for this:

- Crosslinking and non-crosslinking resins tend to yield significantly different hand even for identical backbone compositions.
- Latex particle size affects polymer distribution in the web, which in turn, affects the hand.
- Polymer molecular weight distribution, the surfactant system chosen (especially the surface migration characteristics of the surfactant in the film) and the homogeneity of polymer also play an important role for determining the hand.

To make matters more complex, the method of application of the polymer on the fabric has also been shown to display a great influence on the web properties. For instance, a substantially stiffer (higher  $T_g$ ) polymer has to be used in coating (single – side application technique) than in padding (dual side application) to get equivalent hand. The temperature conditions employed for drying and curing also have a decisive influence on the hand.

#### 2.2 ACRYLIC POLYMERS

Acrylic polymers, being less sensitive to water than starches and VAM-based systems, impart a stiffness that is more durable to washing. Their superior elongation at break also reduces the tendency of the finish film to develop surface cracks. A small quantity of acidic monomer in all acrylic systems not only enhances the adhesion to various fabrics but also often improves stiffness. However, such acrylic products containing acidic units become soluble in alkali and are therefore used primarily in sizing applications. They can then be easily removed during alkali de-sizing.

# 2.3 VINYL ACETATE AND POLYMERS BASED ON VINYL ACETATE

**Vinyl Acetate** CH<sub>2</sub>=CH-CO-CH<sub>3</sub>, is a colorless flammable liquid with an initially pleasant odor that quickly becomes sharp and irritating. It's main use is as a monomer for making poly vinylalcohol and vinyl acetate co-polymers which are widely used in water based paints, adhesives, paper coating or non-woven binders. Poly vinylacetate is the precursor for poly vinyl alcohol and poly vinylacetal resins. Vinylacetate is also co-polymerized as the minor constituent with vinyl chloride or ethylene to form commercial polymers and with acrylonitrile to form acrylic fibers.

Property	Value	Ref. No.
Boiling point ( <sup>0</sup> C)	72.7	16-19
Melting point ( <sup>0</sup> C)	-100, -93	16-19
Specific gravity	0.9338	16-19
Refractive Index	1.3952	16-19
Viscosity at 20 <sup>°</sup> C (poise)	0.42	16-19
Heat of vaporization at 72°C J/g	379.1	16-19
Heat of combustion kJ/g	24.06	16-19
Heat of polymerization kJ / g mol	89.12	18,20
Critical temp. ( <sup>0</sup> C)	252	21
Critical pressure, KPa	4200	21
Critical density g/cm <sup>3</sup>	0.324	21

### **Table-2.1 Physical Properties of Vinyl Acetate**

Vinyl acetate is completely miscible with organic liquids but not with water. At  $20^{\circ}$ C, a saturated solution of vinyl acetate in water contains 2.0-2.4 wt %. Vinyl acetate whereas a saturated solution of water contains 0.9-1.0 wt% water. At  $50^{\circ}$ C, the solubility of vinyl acetate in water is 0.1 wt.% higher than at  $20^{\circ}$ C, but the solubility of water in vinyl acetate doubles to wt %. Vinyl acetate is soluble in a dilute (2.0 wt %) solution of sodium dodecyl sulphate to the extent of 4.0 wt. % at  $30^{\circ}$ C.<sup>22</sup> Azotropes containing vinyl acetate are shown in table.<sup>23</sup>

Second component Azotropic boiling point <sup>0</sup> C		Vinyl acetate wt %
Water	66.0	92.7
Methanol	58.9	63.4
2- propanol	70.8	77.6
Cyclohexane	67.4	61.3
Heptane	72.0	83.5

 Table-2.2 Azotropes Containing Vinyl Acetate

Organic and inorganic peroxides; azo compounds, redox systems, many of which include organomettalic components, light and high-energy radiation, can initiate polymerization. Polymerization is inhibited or strongly retarded by aromatic, hydroxyl nitro or amino compounds and by oxygen, quinone, crotonaldehyde, coppers salts, sulfur. Pure vinyl acetate does not absorb UV significantly at wavelengths large than 250 nm in ethanol or longer than 253 nm in hexane. UV absorption rapidly ends at shorter wavelengths.<sup>24-25</sup>

**Poly Vinyl Acetate** emulsions are widely used as textile finishes because of their low cost and god adhesion to neutral and synthetic fibres. In piece goods finishing, dispersions diluted with water containing 1-3 wt % resin are most often used to obtain a stiff or crisp hand on woven cotton fabrics. Concentration of 2-20,wt. % emulsions is recommended for bodying, stiffening and bonding. Principal applications include the stiffening of felts and the binding of non-

woven fabrics. Finishes to improve snag resistance and body or hands of nylon hosiery are based on PVAc emulsions. The emulsion formulations are usually applied to the cloth by padding form a batch and squeezing off the excess. A formulation can be modified in the pad box to adjusted firmness, by adding an emulsion of softener. The alkali soluble vinyl acetate can be used as warp sizes during weaving. The use of vinyl acetate copolymers as binding agents for non woven fabrics has grown rapidly.

Poly vinyl acetate vary with increasing molecular weight form viscous liquids and low melting solids to hard, brittle polymeric materials. They are neutral, water white to straw-colored, tasteless, odorless, nontoxic products. The resins have no sharply defined melting points but become softer with increasing temperature. They are soluble in organic solvents e.g. esters, ketons, aromatics, halogeneted hydrocarbon, carboxylic acid but insoluble in the lower alcohols (excluding methanol), Glycols, water and non polar liquids.

Absorption of water at 20 <sup>°</sup> C for 24 hr.	3-6%
Cohesive energy density	18.6-19.09
Compressibility	17.8x10 <sup>-6</sup>
Decomposition ( <sup>0</sup> C)	150
Glass transition temp. Tg. ( <sup>0</sup> C)	28-31
Heat capacity at 30 <sup>o</sup> C	1.465
Tensile strength, MPa	29.4-49.0
Young's Modulus, MPa	600
Heat distortin point ( <sup>0</sup> C)	50
Softening temperature ( <sup>0</sup> C)	35-50
Elongation at break at 20 <sup>°</sup> C, MPa	10-20
Modulus of elasticity	1.275-2.256
Thermal conductivity	159
Impact strength, MPa	102.4

# Table-2.3 Physical Properties of Poly Vinyl Acetate (PVA<sub>C)</sub>

Property	Range
Solid (%)	48-55
Viscosity (Poise)	200-4500
pH ( at 27 <sup>°</sup> C)	4-6
Residual monomer % max	0.5
Particle size µm	0.1-3.0
Particle charge	Neutral or negative
Density at 25 <sup>°</sup> C, g/cm <sup>3</sup>	0.92
Stability to borax	Stable or unstable
Mechanical stability	Good or excellent

**Table-2.4 General Emulsion Specification of PVAc** 

By itself, the base polymer is hard ( $T_g \sim 27$  <sup>0</sup>C), but softens on heating above ambient temperatures. The pendant acetyl group makes it sensitive to alkaline hydrolysis whereby it is gradually removed on laundering. Thus, at best PVOAc delivers semi permanent stiffening that improves appearance and aesthetic appeal of the fabric for sales purposes. Due to its good adhesion properties to natural as well as synthetic fibers, it is useful for producing adequately stiff bonded assemblies of different fabrics as well as non-woven structures. Another way<sup>26</sup> to enhance dimensional stability and durability in PVOAc-finished fabric is to add small quantities of rigid thermosetting resins such as ureaformaldehyde and melamine – formaldehyde into the finishing system. In general, PVOAc-based products with a  $T_g \sim 30^{\circ}C$  result in a very stiff film while those having  $T_g$  below  $-10^{\circ}C$  (brought down form the homopolymer  $T_g$ levels by plasticizers or co-monomers) are generally fairly soft. Products with a  $T_g$  of the order of  $-40^{\circ}C$  are very soft, tacky and extensible.<sup>15,27</sup>

Vinyl Acetate / Crotonic Acid copolymers have also been reported to impart stiff hand.<sup>28</sup> However, these are soluble in alkali and do not offer wash fastness. If the same could be made insoluble in alkali by certain means, it could impart a stiffness coupled with durability.

## 2.4 STYRENE-BUTADIENE EMULSIONS IN TEXTILE INDUSTRIES

Emulsion Polymers based on the styrene-butadiene copolymer backbone, with or without additional monomers such as acrylonitrile ("nitrile latex"), 2vinylpyridine ("VP latex") etc., find use not just in textiles, tire cord fabric and carpets but also across a wide spectrum of applications including construction, paper coating and manufacture of latex gloves. Such lattices fall into two broad categories: Carboxylated and Non-carboxylaed. Non-carboxylated latex systems are used mainly for tire cord fabric application and will not be dealt with further here, but a useful article on the subject can be found in Ref. (19). In general, non-modified, non-carboxylated lattices have limitations in adhesion performance because of the non-polar nature of the polymer.

### **Carboxylated Latices**

Once introduced, the carboxyl functional group confers important advantages such as the ability to use sulphurless curing systems (in vulcanization applications), cross linking with other functional monomers and high adhesive strength. Increased polarity of the polymer enhances its compatibility and affinity for polar substrates such as inorganic fillers and fibers. Additional advantages are that incorporation of carboxyl groups improves the mechanical stability of the latex because of electrostatic repulsion between these anionic moieties, which in turn enhances the filler acceptance of the latex.

### Choice of vinyl acids for carboxylated latex systems.

The choice of vinyl monomer acid depends on a number of factor such as

- (a) water-solubility : Some monomers are almost water insoluble e.g. styrene, butadiene etc. and some others are water-soluble e.g. acrylic, methacrylic acids etc.
- (b) End use application: Different vinyl acid monomers are chosen for different end use applications based on historical benefits evolved through the 50 odd years of commercial use. The proportion and chemical structures of the acids selected vary depending on the specific need, for instance acrylic acid is used for paper coating and methacrylic acid in textile finishing.
- (c) Economics: Certain acids such as itaconic acid and crotonic acid provide additional stiffness and dimensional stability to the film formed upon application of latex to the substrate. This is true not just in SBR lattices, but also in emulsion polymers based on other monomers such as vinyl acetate or

acrylates. Such carboxylic acids find utility in specialty carpet backing and textile finishing systems that can bear the higher cost of these monomers.

To obtain the complete advantage of carboxyl groups. it is often necessary that the Carboxylic acid constituents should be introduced into the polymeric backbone either as a pendent group in the straight hydrocarbon chain or as a buried structure on the polymer moiety. The preferred route is copolymerization of a carboxylic acid monomer with other monomers, such as butadiene and styrene. The latex mixture usually contains less than 10 percent of the crboxylic acid monomer. An alternate method for the preparation of the carboxylated styrene butadiene latex is the grafting of the carbxylated monomer on the styrene butadiene latex by using a redox initiator.

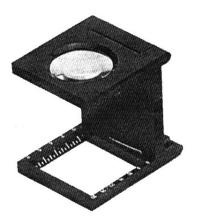
### Chapter 3

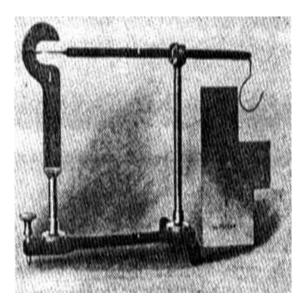
# **TOOLS FOR QUALITY CONTROL**

Here the test procedures along with the necessary equipments used for the quality control are given. The quality control tests, which are relevant to different finishing technologies are given below.

### **GENERAL TESTS FOR FABRIC ANALYSIS**

- 1. Fabric weight per sq. meter
- 2. Ends and pickes per inch (Fig. 3.1)
- 3. Yarn count: Beesley Balance (Fig. 3.2)





### Fig.-3.1 Ends & Pikes Counter

**Fig.-3.2 Beesley Balance** 

### **Desizing: Iodine Staining Test**

This test is based on the development of blue colour when iodine solution is spotted on cotton fabric containing starch. Putting a drop of iodine solution on we desized garment and notice the colour at spotted pattern.

Light yellow to Brown	Complete removal of starch
Violet to Deep Blue	Incomplete desizing
Iodine solution contain :	5 gm potassium Iodine ,1 liter distilled water
	5 gm of iodine in above solution

### **Tests for Different Finishing Processes:**

### a) **Fabric Stiffness:**

This property is the measure of the change in hand or feels of the fabric after giving finishing treatment. Some finishes are given to deliberately make the fabric stiff such as starch finish, or by using synthetic stiffening agents. In some finishes such as in wrinkle free finishing the appliertion of cross linking agent increases the stiffness of the fabric which is undersible. In such cases it is necessary to measure the increase in stiffness of fabric compared to unfinished fabric. The measurement of the fabric stiffness is carried out by the determination of bending length on the instrument as known as "*Stiffness Tester*". Higher the value of the bending length (B/L), greater the fabric stiffness.

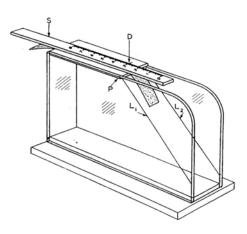


Fig.- 3.3 Stiffness Tester

### b) Crease Recovery Angle

It is the ability of a fabric to recover form the improved deformations. This property given the ability of the fabric to remain free from creases on washing and during use.



Fig.- 3.4 Crease Recovery Tester

### c) Tensile Strength

The tensile strength in the measure of the force required to break the sample when it pulled along the length of the test sample. It is popularly used both for quality control and as a performance standard. Mostly for this purpose "*Instron Tensile Strength Tester*" is used.

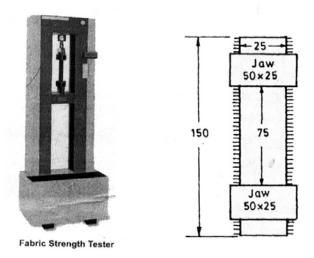
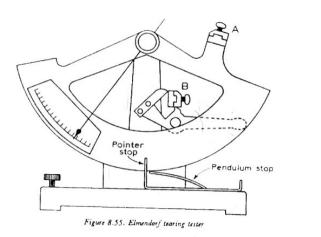


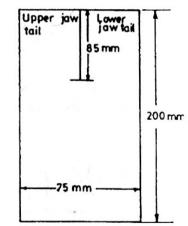
Fig.-3.5 Instron Tensile Strength Tester

### d) Tear Strength

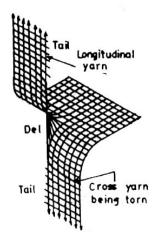
The tear strength of a fabric refers to its resistance to tearing force. Resistance to tearing is of importance in clothing fabrics such as those used for shirting, blouses, interling, and so on and in technical fabrics such as those used for parachutes,. The measurement of tear strength carried out by "*Elmendorf* 

*Tester*" the "Elmendorf" test utilizes a specially designed pendulum tester which measures tear energy.





Tongue tear test specimen



Failure mechanism at tear

**Fig.-3.6 Elmendorf Tester** 

### e) Abrasion Resistance of Fabric

Abrasion is the wearing away of any part of a fabric by rubbing against another surface. Thus the abrasion resistance of fabric is the ability to withstand the rubbing (frictional force) applied to its surface. This test is carried out on instrument known as "*Martindale Abrasion Tester*".

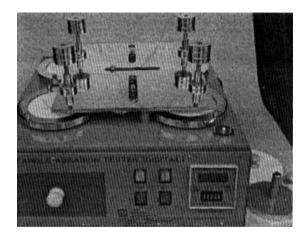


Fig.-3.7 Martindale Abrasion Tester

# Chapter 4 EXPERIMENTAL DETAILS

A number of experiments were carried out to develop correlation between the handle and tensile properties of the film and the handle of fabric with the glass transition temperature  $(T_g)$  of deposited polymer. My objective is to investigate the structure property relationships in textile finishing.

Acrylic polymers, Polyvinyl Acetate and its derivatives and copolymers and styrene – Butadiene emulsions etc. were used as functionalized polymer for textile treatment. In case of styrene – Butadiene it may be modified or non-modified i.e. Carboxylated or non-Carboxylated). This increases polarity and thus compatibility and affinity of inorgnaic fillers and fabrics also increases. Carboxylated styrene – butaliene may be used with Vinylmonomer.

### Approach

With a view of structure – property relationships and to develop more efficient and durable stiffeners two stage approach was taken up using Polyvinyl Acetate system. The objective of the 1<sup>st</sup> stage was to identify the factors that could theoretically affect the hardness of this resin and then conduct a systematic study using a *Statistical Experimental design technique*. In the second stage, a few other chemicals included which are commonly employed in the stiffening / finishing recipes mainly with a view to enhance the stiffness / durability / smoothness.

Polyvinyl acetate homopolymer of 50% solid was mixed with DBP, PE and Sarch solution (14%). DM water was added to maintain the solid content of the

finished bath composition constant. 20-part emulsion and 80-part water were taken. After taking the weight of the fabric, it was dipped in the solution and then padded in the padding mangle at a speed of 40 rpm and 1 kg/cm<sup>2</sup> pressure. After padding, the fabrics were dried at  $110^{\circ}$ C for five minute. The eight of the finished fabric was taken and bending length was measured.

### Materials

Cotton

	Warp Count	88 per inch
	Weft count	92 per inch
Polyester		
	Warp Count	92 per inch
	Weft count	94 pe rinch

### Base System

Vinyl Acetate, Additives ,Polyvinyl alcohol, Cross linker ,Dibutyl pthalate, PE Emulsion, Starch etc.

### Procedure

In the first stage of work, total 9 products based on Polyvinyl Acetate System were made. The obtained material was used for finishing cotton fabric. The finished fabrics were subjected to measurement of bending length, body and similarly hardness of film on glass plate at ambient temperature.

In second stage, both cotton as well as 100% polyester fabrics were included. For finishing purpose step-by-step procedure is given below:

- A solution of 40 gm per lit was made of selected emulation stirred well, for complete dissolution of emulsion.
- 2. Substrate (fabric) of specific size was take and weighted for percentage addition calculation. Fabric was dipped properly.
- 3. Now substrate cloth was padded in the padding mangle at a speed of 40 rpm and 1 kg/cm<sup>2</sup>.
- 4. After padding, the fabric is dried at  $110^{\circ}$ C for 5 minutes. And then cured at  $150^{\circ}$ C for 3-5 minutes.
- 5. After this weight and bending length of finished fabric was taken.

# Chapter 5 RESULTS AND DISCUSSION

The study was carried out in two stages. In the 1<sup>st</sup> stage poly vinyl acetate of high level of hardness was prepared. For this three factors are chosen namely % of poly vinyl alcohol, % of initiator and % of cross linker. Total nine products were made.

Ex. No.	% of PVOH*	% of Initiator	% of Cross linker
1	5.0	0.18	0.0
2	5.0	0.20	1.0
3	5.0	0.22	2.0
4	6.5	0.18	1.0
5	6.5	0.20	2.0
6	6.5	0.22	0.0
7	8.0	0.18	2.0
8	8.0	0.20	0.0
9	8.0	0.22	1.0

Table 5.1 Details of Product to be Formed

\* PVOH – Polyvinyl Alcohol

### Table 5.2

Ex. No.	Solid (%)	Viscosity (Ps)	pH (at 27ºC)	Film Properteis	Borax Stability	No. of average Mol. Wt. (Mn.)
1	51.22	460	5.17	Clear	Unstable	31,749
2	51.00	440	5.09	Clear	Unstable	32,930
3	49.70	380	5.16	Clear	Unstable	23,055
4	51.60	700	5.14	Clear	Unstable	30,471
5	50.56	760	5.01	Clear	Unstable	26,955
6	51.00	780	5.02	Clear	Unstable	27,977
7	50.20	760	5.20	Clear	Unstable	34,324
8	49.00	1040	5.16	Sl. Hazy	Unstable	25,292
9	49.56	770	5.11	Clear	Unstable	23,462

# Analytical Data of the Obtained poly(vinyl acetate)

Finishing was done on cotton fabric and following data were collected for studying stiffness, body and hardness.

Ex. No.	Stiffness	Body	Hardness (% of glass)
1	4.29	2.90	25
2	4.26	3.15	36
3	4.75	3.58	35
4	4.83	3.26	27
5	4.77	3.33	26
6	4.75	3.45	25
7	4.88	3.60	38
8	4.79	3.24	18
9	4.66	3.52	28

 Table-5.3 Analytical Data on Application Performance on Cotton

In the  $2^{nd}$  stage another set of experiments were carried out by varying the concentration level of three chosen additive ingredients. Each of these ingredients was attempted at two levels of incorporation.

Table -	-5.4
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Factors	Level 1	Level 2
Dibutyl phthalate	10.00	20.00
PE Emulsion	6.66	13.33
Starch	10.00	20.00

In this stage, both cotton as well as 100% polyester fabric included. The layout of the experiments for preparation of the samples used given below:

Ex. No.	DBP	PE Emulsion	Starch
1	10	6.66	10
2	10	6.66	20
3	10	13.33	10
4	10	13.33	20
5	20	6.66	10
6	20	6.66	20
7	20	13.33	10
8	20	13.33	20

### Table 5.5

The analytical data of stiffness and body on polyester fabric and cotton fabric are given in table

Experiments	Stiff	ness	Body		
no.	Ι	II	Ι	II	
1	2.73	2.66	2.17	4.21	
2	2.58	2.97	3.58	2.37	
3	3.39	3.43	3.93	2.65	
4	2.71	2.64	3.41	4.81	
5	2.45	2.29	3.45	4.72	
6	2.34	2.41	4.54	3.10	
7	2.58	2.43	3.92	3.11	
8	3.05	3.28	3.25	4.74	

 Table-5.6 Analytical Data of Stiffness and Body on Polyester Fabric

#### Table 5.7

Experiments no.	Stiff	'ness	Body		
	Ι	II	Ι	II	
1	2.94	2.94	7.57	7.61	
2	3.14	3.09	8.32	8.11	
3	3.04	3.05	9.45	8.38	
4	2.97	2.63	8.614	8.81	
5	2.96	2.83	8.68	7.65	
6	2.94	2.85	7.64	8.75	
7	2.86	2.85	7.73	8.25	
8	3.12	2.79	9.17	7.45	

#### Analytical Data of Stiffness and Body on Cotton Fabric

From the Table 5.1 - 5.2, it is seen that when employed at 5% concentration polyvinyl alcohol at a concentration of initiator e.g. 0.20% and 0.22% and concentration of cross linking agent e.g. 1.0% and 2.0% the obtained Poly Vinyl acetate shows hardness e.g. 36% of glass and 35% of glass. When the concentration of polyvinyl alcohol is increased form 5.0% to 6.5%, there is no significant effect is seen on the hardness of film. When employing PVOH at higher concentration of 8% and the concentration of initiator and cross linking agent is 0.18% and 0.20% respectively, the results shown hardness and better stiffness and body.

Systematic data on film hardness,  $T_g$  and MFFT has been collected (Table5.8) for "pure acrylic" products i.e. those which contain only acrylate / methacrylate esters and acrylic / methacrylic acids in the backbone monomer composition. Some of these are have a self-crosslinking group, such as allyl methacrylate, while some are cross linkable through additives or non-crosslinking altogether.<sup>13</sup> Such correlations can help identify the right finishing system for a fabric under specified conditions and desired end-results.

Polymer Type	T <sub>g</sub> ( <sup>0</sup> K)	MFFT( <sup>0</sup> K)	рН	Viscosity (cps)	Film properties
Cross	267	<273	3.4	145	Soft
Linkable	274	<273	6.4	50	Soft
	2850	<273	8.50	50	Very soft
Self Cross linkable	263	<273	3.0	550	Soft
	292	278	3.0	550	Full
	307	295	3.5	30	Firm
	308	285	5.5	60	Firm

Table-5.8

In another series of experiments (Table-5.9) we have compared various 45% solid emulsion polymer of containing vinyl acetate, various acrylates and

**styrene** in different proportions. The impact on polymer properties and bending length of treated fabric is shown below (13,14).

Exp. No.	Predicated Tg <sup>0</sup> K	Visc. (cps)	РН	Viscosity (cps) after thickening	Polyester viscose		100% Polyester	
					% Add on	B/L	% Add on	B/L
*PP/01/236	322	65	3.00	700	16.95	5.60	24.12	4.50
*PP/01/176	333	100	2.90	2900	15.68	5.1	20.40	4.25
*PP/01/226	311	70	3.15	1300	15.66	5.2	29.96	4.25
**PRD3/7282/01	303	4000	4.80	Not Practical	16.17	5.75	22.50	4.40
**PRD3/3285/01	288	400	4.00	Not Practical	16.61	5.00	24.50	3.90

### Table 2

\*1-3 are self cross – linking type products,

\*\*4-5 are produced 50% and then diluted to 45%

### Impact of polymer selection

At Jubilant Organosys, they developed a series of polymers of different chemistries and applied them on various fabrics at an add-on level of 2%. The obtained fabrics have been compared in terms of:

- (a) Bending length (BL Table 5.10)
- (b) Crease recovery properties and
- (c) Body / draping characteristics.

The polymer films themselves were studied for hardness (which correlates moderately with fabric results), clarity and tack. While molecular weight determination,  $T_g$  and MFFT measurements were also carried out, *no statistically significant correlations of these parameters to eventual fabric characteristics could be identified*.

S. NO.	Monomers	Crosslinker	Avg. BL/Col (cm.)	Avg BL/PET (cm.)	Hardness (% of glass)
1.	Vam/Mal	No	5.4	5.45	13.3
2.	Vam/Mal	No	5.25	4.9	9.7
3.	Vam	Yes	6.85	4.3	21.9
4.	Vam/Acr	Yes	4.3	3.45	1.9
5.	Vam/Acr	Yes	4.75	5.1	3.8
6.	Vam./Veova	No	5.35	4.9	23.8
7.	Vam/Acr	Yes	4.8	3.85	14.1
8.	Vam/Veova	No	5.5	4.85	15.5
9.	Acr	Yes	5.6	4.5	31.4
10.	Sty/Acr	Yes	4.4	3.45	14.8
11.	Vam/Acr	Yes	5.0	4.25	25
12.	Vam	Yes	6.35	4.15	16.1
13.	Sty/Acr	Yes	4.6	3.65	14.3
14.	Vam.Eth	No	4.6	3.5	4.3

Table-5.10

VAM = vinyl acetate; Mal = maleic esters; Acr = Acrylic esters; VeOVa = vinyl ester of versatic acid; Sty = styrene; Eth = ethylene

### CONCLUSION

Following points are concluded form study:

- 1. The important conclusion from the analysis of experimental result in first stage (i.e. no additive ingredients) is that concentration of protective collide and concentration of initiator were the main factor influencing hardness.
- 2. Effect of additive ingradients depends on the type of fabric to be finished.

For example:

**For cotton** – The interaction of Dibutyl Phthalate and polyethylene emulsion are found to be significant at 5% level. Starch doesn't have any effect.

**For polyester** – The interaction of Dibutyle Phthalate Polyethylene emulsion and diabutyle phthalate and starch significant at 5% level.

- 3. Although film properties are well correlated with polymer  $T_g$ , the handle of web and other properties are not correlated with the binder  $T_g$ .
- 4. Besides the physical properties of the polymer, the temperature employed for drying and curing also have very decisive influence on the hand.
- 5. In case of caboxylated SBR, the effective distribution of carboxyl groups through optimization of polymerization conditions & careful selection of

the emulsifier system results in excellent mechanical stability & compatibility.

- 6. A fine and controlled practical size ensures excellent binding power, required for stabilization & anchoring the individual tuft.
- 7. The proportion & Chemical structure of functionalizing monomer can vary, depending on the specific need.

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