Synthesis Of Fe^{3+} Ion Crosslinked Green Hydrogel Using Xanthan Gum And Sodium Carboxymethyl Cellulose For Agricultural Purpose

A DISSERTATION

SUBMITTED IN THE PARTIAL FULFILLMENT OF THE

REQUIREMENTS FOR THE AWARD OF THE DEGREE OF

MASTER OF SCIENCE

IN

CHEMISTRY

SUBMITTED BY

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May 2023

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CANDIDATE'S DECLARTION

I, Rahul Yadav (2K21/MSCCHE/35) and Ayush (2K21/MSCCHE/09) students of M.Sc. Chemistry hereby declare that the project Dissertation titled "Synthesis Of Fe^{3+} Ion Crosslinked Green Hydrogel Using Xanthan Gum And Sodium Carboxymethyl Cellulose For Agricultural Purpose" which is submitted by us to the Department of Applied Chemistry, Delhi Technological University, Delhi in the partial fulfillment of the requirement for the award of the degree of Master of Science, is original and not copied from any source without proper citation. This work has not previously formed the basis for the award of any Degree, Diploma, Associateship, Fellowship or other similar title or recognition.

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Date: 29/05/2023	**(2K21/MSCCHE/35)**	**(2K21/MSCCHE/09)**

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CERTIFICATE

I/We hereby certify that the Project Dissertation titled "Synthesis of Fe^{3+} ion crosslinked Green Hydrogel using Xanthan Gum and Sodium Carboxymethyl cellulose For Agricultural purpose" which is submitted by Rahul Yadav (2K21/MSCCHE/35) & Ayush (2K21/MSCCHE/09), Department of Applied Chemistry, Delhi Technological University, Delhi in partial fulfillment of the requirement for the award of the Master of Science, is a record of the project work carried out by the students under my supervision. To the best of my/our knowledge this work has not been submitted in part or full for any Degree or Diploma to this University or elsewhere.

Place: Delhi **Prof. Archna Rani**

Date: 29/05/2023 (Supervisor)

ACKNOWLEDGEMENT

The success and final outcome of this project required a lot of guidance and assistance from many people and we are extremely fortunate to have got this all along the completion of this project work.

We wish to express our gratitude towards my project supervisor, **Prof. Archna Rani, Department of Applied Chemistry, Delhi Technological University**, who provided us a golden opportunity to work under their able guidance. Their scholastic guidance and sagacious suggestions helped me to complete the project on time

We wish to thank **Prof. Anil Kumar, Head of the Department of Applied Chemistry**, Delhi Technological University for his constant motivation.

We are thankful to and fortunate enough to get constant encouragement, support and guidance from all teaching staffs of Department of Applied Chemistry, which helped me in successfully completing my project work. We are also thankful to **PhD scholars Ms. ManU, Mrs. Meenakshi Tanwar and Ms. Ritu Sharma** for their constant support and motivation.

Finally, yet importantly, We would like to express our heartfelt thanks to our beloved family and friends who have endured our long working hours and whose motivation kept us going.

Rahul Yadav & Ayush

ABSTRACT

Ever-growing world population is creating immense pressure on the agriculture sector leading to the overexploitation of resources, soil degradation, and water scarcity which in turn reduces crop yield significantly. Current research has been started with the goal of creating new materials for the effective use of water in agriculture in order to stop this damaging cycle. Thus, a series of Xanthan gum (XG) and Carboxymethylcellulose sodium salt (NaCMC) based ionic crosslinked hydrogels (XG- Fe-$NaCMC_1$-XG-Fe-$NaCMC_5$) were synthesized. The macromolecular chain's abundance of reactive hydroxyl and carboxylate groups makes it possible to connect the chains using multivalent ionic crosslinkers for example trivalent iron (Fe^{3+}). The hydrogels were characterized using Fourier-transform infrared spectroscopy, thermo- gravimetric analysis, scanning electron microscopy, and X-ray diffraction analysis. The maximum water-holding capacity of hydrogel-amended soil was observed.

Hydrogel was also studied by pot experiment for the wheatgrass (*Triticum aestivum*) growth study. The study confirms that the hydrogel, XG-Fe-NaCMC with maximum thermal stability and improved swelling index has potential application in agriculture by improving the water holding capacity of the soil and can also be applied to agricultural areas as a fertilizer release system.

Keywords

Hydrogel; NaCMC; Xanthan Gum; Agriculture

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CHAPTER-1

INTRODUCTION

1. Introduction

Agriculture is one of the biggest consumers of water. It causes water scarcity and at the same time, it is badly impacted by it. The unpredictable profile of rainfall and insufficient water retention by soil also contribute to the loss of plant nutrients in modern agriculture[1].The survival of livelihoods depending on water and agriculture is being threatened by the overuse and decrease of water resources. Moreover, in order to feed a population of over nine billion individuals by 2050, agricultural output would need to grow by 50%, and water consumption will need to rise by 15%[2]. To fulfill the increasing food demand and for lowering the rising burden on water resources immediate action must be taken. The use of hydrogels for agricultural practice is one of the accepted solutions to address these problems[3].

Hydrogels are polymeric materials with three-dimensional (3D) network structures having hydrophilic polymer chains[4]. Despite not dissolving in an aqueous media, they do swell in aqueous solutions enough to absorb a significant quantity of water since their polymer networks contain hydrophilic groups including $–NH_2$, –OH, –COOH, $–SO_3H$ in their polymer networks [4,5]. In agriculture, the capacity of hydrogel to absorb and hold substantial volumes of water has become extremely important. They serve as miniature water reservoirs in the soil, discharging water when needed by plant roots. Additionally, there are studies on the beneficial change of soil's physical characteristics by hydrogels [6]. Despite the fact that commercially available products based on polyacrylamide (PAAm) and acrylate derivatives have shown the benefits of using hydrogels in agriculture, these are regarded as soil pollutants since they are not entirely biodegradable. Even while PAAm is not classified as harmful, commercial formulations nevertheless include trace levels of acrylamide, a neurotoxin and carcinogen that raises questions about potential soil and food contamination. As a result, hydrogels made of biopolymers have become increasingly popular because of their environmental friendliness and ability to break down naturally . Also, they are less expensive than synthetic ones. Among numerous natural polymers, the preference is given to those which have functional groups that make them easy to modify[7].

Xanthan Gum(XG)(Fig.1.) and Cellulose are examples of natural polymers. These polymers form an ideal source of hydrogel synthesis because of their biocompatibility, and non toxicity properties. XG is a well-known water-soluble polysaccharide formed by the fermentation of glucose, sucrose, or lactose via the bacteria Xanthomonas campestris, and it is a useful food hydrocolloid. It is widely utilized in the food sector as food additives [8]. Cellulose, is a naturally derived polysaccharide. It is generally modified by combining with sodium mono chloroacetate in an alkaline media producing Carboxy methyl cellulose sodium salt (NaCMC). NaCMC(Fig.2.) is a polyelectrolyte, thus considered a "smart" cellulose derivative, which is sensitive to pH ,water soluble and ionic strength fluctuations. The polymer chains have a large number of reactive hydroxyl groups. Additionally, the presence of carboxylate groups in the polymeric chain enables multivalent ionic crosslinking to bind the chains together [9,10].The hydrogel networks are closely linked through hydrophobic connections, hydrogen bonds, and ionic coordination interactions between −COO− of XG chains, NaCMC chains, and Fe^{3+} ions[11].Ionic hydrogels may be created using a variety of ions. Trivalent iron (Fe^{3+})is a good ion to use for creating XG-Fe-NaCMC hydrogels since it is a safe and non-toxic cross linking agent[10]. Owing to its benefits, composites of Xanthan Gum and cellulose fibers were developed to be used both as soil conditioners and topsoil covers, to promote plant growth and forest protection by Sorze et al.[12]. Akalin et al. and Ahuja et al. used NaCMC for the preparation of hydrogel.

The current study is focussed on XG-Fe and NaCMC-Fe hydrogels to potential use in agriculture of XG-Fe-NaCMC hydrogel with XG and NaCMC-based hydrogels. As per our best knowledge, no such work has been done so far. Various XG and NaCMC based hydrogels have been reported in different types of applications. After detail survey of this literature, it found that natural products are more efficient to save our environment but petro-based sources are dominating one, which have been used on large scale. So if we want to produce a green hydrogel, we have to use a nontoxic crosslinker. To fill this gap we introduced $FeCl_3$crosslinker which is totally helpful for plants as they need iron too. We processed our work by addition XG in NaCMC and fabricated a hydrogel using XG/NaCMC crosslinked with $FeCl_3$ so that its swelling can be increased and it will be useful for agricultural purpose. No work of this type has been done yet. Along with XG-Fe and NaCMC-Fe also fabricated for the comparison from synthesized novel XG-Fe-NaCMC hydrogel. The swelling test was carried out to identify the optimum XG-Fe-NaCMC hydrogel preparation parameters. In addition, Fourier Transform Infrared (FTIR), Scanning Electron Microscope/Energy Dispersive X-ray Analysis (SEM/EDX), and Thermal Stability through TGA were used to characterise the chemical structures, surface morphologies, elemental compositions, and surface roughness parameters of hydrogel. The synthesised hydrogel was studied agriculture use by the plant growth study. Wheatgrass has been grown for 15 days and the difference in total fresh and dry weight along with average root and shoot height have been reported. The improvement in plant performance caused by utilizing these hydrogels suggests that they can be employed in agricultural fields.

CHAPTER-2

LITERATURE REVIEW

2.1 Natural Sources:

2.1.1 Xanthan Gum(XG):

The first organic biopolymer created on an industrial basis is xanthan gum. XG is a water soluble well known polysaccharide produced by the fermentation of glucose, sucrose, or lactosevia bacteria Xanthomonas campestris, is a useful food hydrocolloid. It is extensively used in food industry because of its biocompatibility, biodegradability, non-toxicity, and low cost. It has a cellulose-type main-chain (1 / 4)-β-glucan with trisaccharide side-chains attached to alternate glucose units in the main-chain 17 and has been widely used in a broad range of industries as a rheological control agent in aqueous systems and as a stabilizer for emulsions and suspensions[11,13]. This gum's safety has been thoroughly studied. In oral animal trials, the acute toxicity of xanthan gum was assessed. There was no toxicity that could be noticed for xanthan gum concentrations up to 20 g/kg body weight. experimented by XG has more stable thermal behavior against hydrolysis than other water-soluble polysaccharides because of its helical structure ordered form that protects XG molecules from

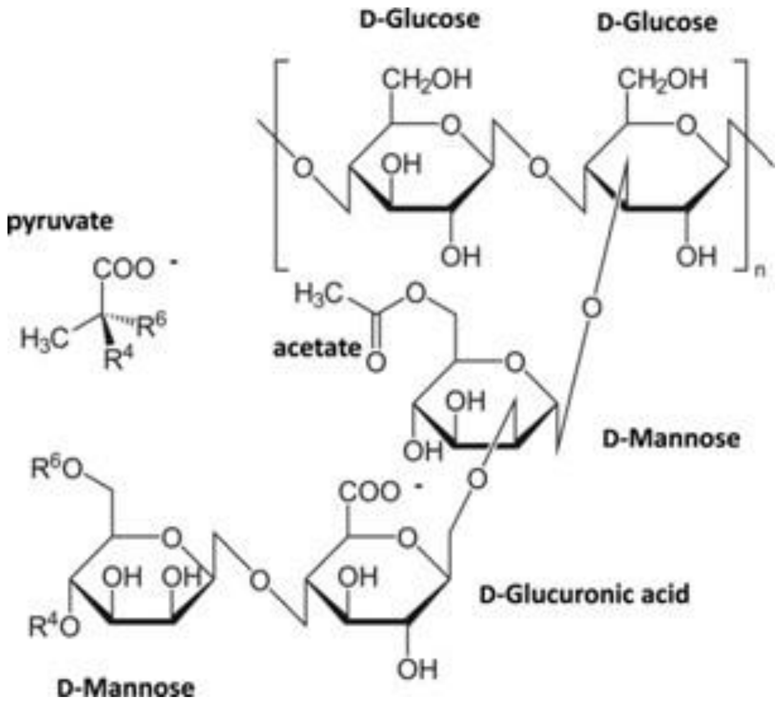


Fig. 1, Structure of Xanthan Gum

depolymerisation[14].

2.1.2 Sodium carboxymethyl cellulose (NaCMC):

NaCMC is a non-toxic, biocompatible, bioadhesive, biodegradable and water soluble natural polysaccharide[9].It is produced by reacting sodium monochloro-acetate with cellulose in alkaline medium. It is also known that NaCMC is a polyelectrolyte, and thus this "smart" cellulose derivative presents sensitivity to pH and ionic strength variations. It is easy to form NaCMC hydrogels because of the large number of reactive hydroxyl groups on the polymer chains. Besides, the presence of carboxylate groups in the macromolecular chain enables bonding the chains to each other via multivalent ionic crosslinking[15].

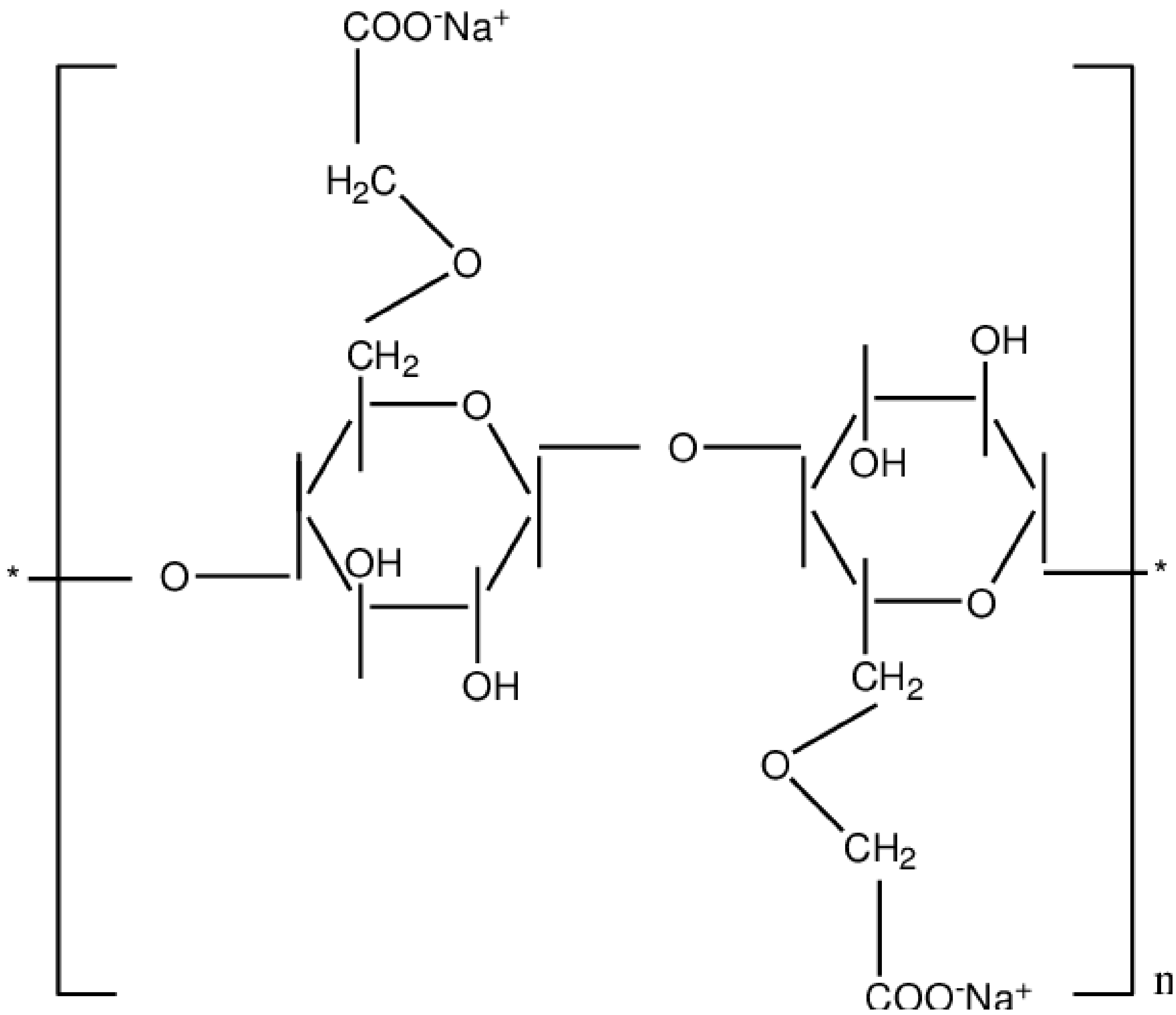


Fig. 2, Structure of Sodium carboxymethyl cellulose

2.2 Classification of hydrogels:

Hydrogels are categorized based on their physical characteristics, kind of swelling, manufacturing process, origin, ionic charges, sources, rate of biodegradation, and type of crosslinking that has been identified[14]. In general, either synthetic or biological components can be used to create hydrogels. Petroleum-based goods are found in synthetic polymers, whereas natural sources including cellulose, starch, lignin, and kenaf fibre are found in bio-based hydrogels. In comparison to hydrogels made from biological materials, synthetic hydrogels are less hydrophilic and mechanically more resilient[16].

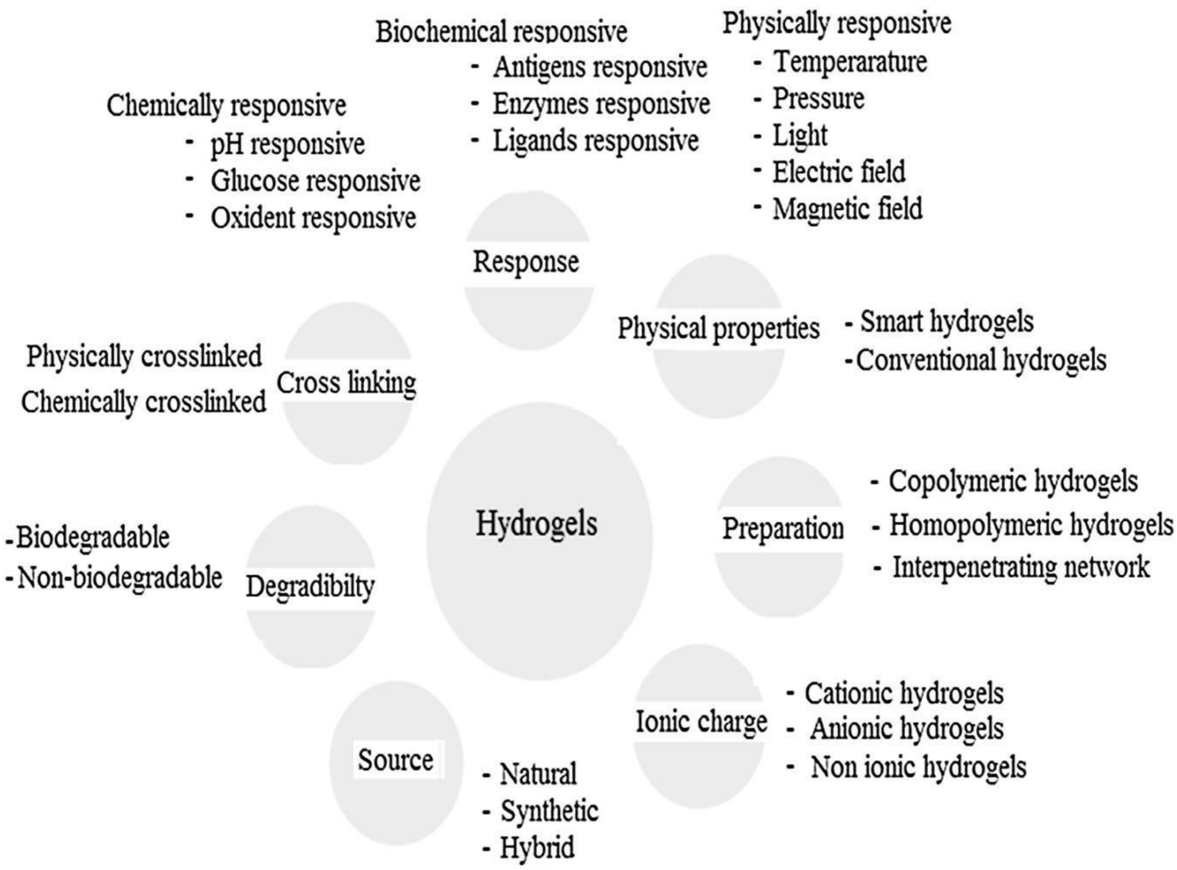


Fig. 3, Classification of hydrogels

2.2.1 Classification based on sources:

Polymers that are created synthetically or organically can be crosslinked to create hydrogels. In the beginning, synthetic hydrogels including polyacrylamide, polyvinyl alcohol, and polyethylene oxide were utilised for agricultural applications[17]. However, it was subsequently discovered that these hydrogels might be hazardous to the environment [18]. Synthetic hydrogels have a high swelling degree, a high level of resilience, and a moderate rate of degradation However, hydrogels made from natural polymers have a weak structure and little swelling. According to Ahmadi et al. hydrogels made from natural polymers can be biodegrade.[17] The breakdown products can occasionally even make the soil more fertile. A novel class of hydrogels may be created by crosslinking natural polymers onto synthetic polymers in order to combine the benefits of both natural polymer-based hydrogel and synthetic hydrogel.[19]

2.2.2 Classification based on crosslinking:

The capacity of the polymers to produce elastic three-dimensional matrices that allow for swelling while also stabilizing the matrix's structure is entirely due to crosslinking. It is possible to crosslink hydrogels both chemically and physically via ionic interactions, hydrogen bonds, and hydrophobic interactions, as well as polymerization, graft polymerization, network creation of a water-soluble polymer, and radiation crosslinking. Physically and chemically crosslinked hydrogels differ in that the former have permanent links while the latter have transitory ones . The hydrogel gains strength and durability through chemical crosslinking, which is irreversible. For applications involving high temperatures, fluctuating pH levels, and pressure, this characteristic is helpful. physical cross-linking is reversible, Because of its fast reaction to destabilizing under external stimuli in the environment[19].

2.3 The Importance of Cross-linking:

Adding cross-linkers between polymer chains changes the polymer's physical characteristics depending on the degree of cross-linking as well as whether crystallinity is present or not[20]. The hydrogel gains the following enhanced characteristics as a result of cross linking:

- Decreasing viscosity (the barrier to flow) and elasticity (the capacity to stretch and return to their former form)
- Insolubility
- expanded toughness and strength, and a lower melting point
- Thermoplasts are converted to thermosets

2.4 Characterstics of Hydrogels:

Hydrogels are rare substances because of their ability to absorb water and their flexibility. Hydrophilic functional groups connected to the polymer backbone provide hydrogels their capacity to absorb water, while cross-links between network chains provide their capacity to resist dissolution[4,24]. The following is a list of characteristics of excellent hydrogel materials:

- Hydrogels are non-toxic materials that are colourless and odourless.
- They have a great capacity for absorbing water.
- The least soluble component and the remaining monomer.
- Greater durability and consistency.
- Biodegradability
- Upon swelling in the water, pH returns to neutral.
- Good permeability.
- Re-wetting capability

2.5 Applications of Natural sources based Hydrogels:

Hydrogels are a significant group of materials with remarkable applications in agriculture, engineering, biology, food industry, and pharmaceutical sciences. Considering that opposite-charged species may bind with polyelectrolyte hydrogels to form complexes and either carry or create charges on the chain, polyelectrolyte hydrogels are particularly beneficial[22].

Fig.4. Applications of natural polymer based hydrogel

2.6 XG-based chemically crosslinked hydrogels:

Three methods may be used to create chemically crosslinked hydrogels utilizing XG. Crosslinking agents including sodium trimetaphosphate, citric acid, and glycerol are used in the first route.The second, however, is accomplished by crosslinking agents and starting materials during the polymerization of vinyl monomers such as acrylic acid, vinyl imidazole, and vinyl pyridine on the XG backbone[23].The third one has to do with the crosslinking of XG when water-soluble polymers are present. For instance, Bejenariu et al. created chemically crosslinked XG hydrogels using just the XG backbone[24]using a non-toxic cyclic crosslinker (sodium tri metaphosphate, STMP) in an alkaline medium in accordance with the solvent-free alkaline technique.

2.7 XG-based physically crosslinked hydrogels:

XG-based physically crosslinked hydrogels can be created by a variety of interactions, including ionic (electrostatic), hydrogen bonds, and hydrophobic force interactions.In particular, freeze-thawing is one of the methods that may produce hydrogels without the usage of crosslinking agents since phase separation happened for the polymer solutions. Additionally, the interpenetrating polymer network (IPN) is primarily created to enhance the mechanical characteristics and swelling behavior of hydrogels.[24]

Table.1.Reported XG based hydrogels and their applications.

Source	Crosslinker	Applications	References
XG, Starch	Sodium trimethaphosphate (STMP) and sodium sulphate	Controlled drug delivery	[25]
XG/MMT, Acrylamide (AAm), acrylonitrile (AN)	$FeCl_3$	wound dressings	[11]
XG-Poly(vinyl alcohol)	H- Bonding	-	[26]
XG, APS, 2-Hydroxyethyl methacrylate	MBA (N,Nmethylenebisacrylamide)	Biomedical	[27]
XG, Maleic anhydride,		Dye Removal	[28]

DMF			
XG, Alignate	$CaCl_2$	Wound healing	[29]
XG, Poly acrylic acid	N, N′-hexane-1, 6-dilbisprop-2-enamid (MS)	Drug release	[30]
XG, Starch	Citric acid	food and drug areas.	[31]

2.8 NaCMCbased chemically crosslinked hydrogels:

NaCMC based chemically crosslinked hydrogels can be created by various methods like free radical initiator, irradiation and enzymatic reactions. Hydrogel formed by chemical crosslinking is more stable and permanent as they do not dissolve in water without cleavage of bonds[32]. NaCMC chemical crosslinked hydrogel can be made by crosslinked with citric acid, acrylic acid[33].

2.9 NaCMC-based physically crosslinked hydrogels:

NaCMC based physically crosslinked hydrogels can be created by a variety of interactions, including ionic (electrostatic), hydrogen bonds, and hydrophobic force interactions .Freeze-thawing is one of the methods that may produce hydrogels without the usage of crosslinking.(PVA/NaCMC) hydrogels were prepared by physical crosslinking (cyclic freezing/thawing)[34].

Table.2.Reported NaCMC based hydrogels and their applications.

Source	**Crosslinker**	**Application**	**References**
NaCMC, HPMC	Citric acid	Antimicrobial Activity, drug delivery and wound dressing	[35]
NaCMC	$FeCl_3$	Bio Materials	[10]
NaCMC	$CaCl_2$	biodegradable sorbent materialanddrug carrier	[35]
NaCMC	Fumaric acid	Wound Dressing	[36]
NaCMC	PVA	Wound Healing	[37]
NaCMC	$FeCl_3$	treatment of agricultural drainage	[38]
NaCMC, graphene oxide	$FeCl_3$	Drug release	[39]
NaCMC	Boric acid	Pesticide release in agriculture	[40]
NaCMC and carrageenan	$FeCl_3.6H_2O$	Plant growth	[41]
NaCMC with nanocellulose	Citric acid	reduce fertilizer loss & improve water use in agriculture.	[42]

CHAPTER-3

MATERIALS AND METHODS

3.1 Chemicals:

XG powder was purchased from the local market, Iron (III) chloride ($FeCl_3$) was purchased from Merck Life Sciences Pvt. Ltd**,** NaCMC(Sodium Carboxy methyl cellulose)was purchased from CDH Pvt. Ltd. Urea was purchased from CDH Pvt. Ltd. Milli Q grade water was used to prepare aqueous solutions.

3.2 Synthesis of XG- Fe-NaCMC hydrogel:

A series of three sets of hydrogels was prepared named XG-Fe, NaCMC-Fe, and XG-Fe-NaCMC (here XG-Fe represents Xanthan Gum based hydrogel, NaCMC-Fe represents the hydrogel of Sodium salt of Carboxymethyl Cellulose and XG-Fe-NaCMC is hydrogel of mixture of Xanthan gum and Carboxymethyl Cellulose).

For preparation of all three sets a constant weight of xanthan gum and carboxymethyl cellulose was taken (i.e 0.4 g XG for XG-Fe ,0.4g NaCMC for NaCMC-Fe and 0.2g XG& 0.2g NaCMC for XG-Fe-NaCMC) and dissolved in 40ml of distilled water with the help of magnetic stirrer till a homogeneous solution was obtained. Different concentration of aqueous $FeCl_3$ solution (0.4g – 0.2g) per 40ml were prepared for crosslinking. The homogeneous solution mixture of all three sets were soaked with crosslinker aqueous $FeCl_3$ and left for 48 hours at room temperature for uniform crosslinking between Fe^{3+} ions and carboxyl groups of Xanthan gum and Carboxymethyl cellulose. After 48 hours hydrogel formed was soaked in distilled water for 24 hours for removal of any unreacted material left. The hydrogel was then oven dried at a temperature of 70°C and powdered with help of mortar – pestle for characterization and application processes.

Table.3. Composition of XG and NaCMC inXG-Fe with various $FeCl_3$ concentrations

Formulation	Xanthan Gum(g)	NaCMC(g)	Distilled Water(ml)	Crosslinker-$FeCl_3$(g/40 ml)	Swelling Index(%)
XG-Fe_1	0.4	0	40	0.04	NA*
XG-Fe_2	0.4	0	40	0.08	702.1
XG-Fe_3	0.4	0	40	0.12	521.5
XG-Fe_4	0.4	0	40	0.16	479.7
XG-Fe_5	0.4	0	40	0.20	413.5

Table.4.Composition of XG andNaCMC in NaCMC-Fe with various $FeCl_3$ concentrations

Formulation	Xanthan Gum(g)	NaCMC(g)	Distilled Water(ml)	Crosslinker-$FeCl_3$(g/40 ml)	Swelling Index(%)
NaCMC-Fe_1	0	0.4	40	0.04	NA*
NaCMC-Fe_2	0	0.4	40	0.08	364.8
NaCMC-Fe_3	0	0.4	40	0.12	343.2
NaCMC-Fe_4	0	0.4	40	0.16	241.4
NaCMC-Fe_5	0	0.4	40	0.20	231.3

Table.5.Composition of XG and NaCMC in XG-Fe-NaCMC with various $FeCl_3$ concentrations

Formulation	Xanthan Gum(g)	NaCMC(g)	Distilled Water(ml)	Crosslinker-$FeCl_3$(g/40 ml)	Swelling Index (%)
XG-Fe-CMC_1	0.2	0.2	40	0.04	NA*
XG-Fe-$NaCMC_2$	0.2	0.2	40	0.08	526.4
XG-Fe-$NaCMC_3$	0.2	0.2	40	0.12	412.2
XG-Fe-$NaCMC_4$	0.2	0.2	40	0.16	360.3
XG-Fe-$NaCMC_5$	0.2	0.2	40	0.20	310.2

Hydrogel having 0.08g/40mL $Fecl_3$ is the highest swelling index among all sets. So this is considered as an optimum formulation for further studies.

NA* represents that the hydrogel is not formed for that formulation.

3.3 Swelling Study:

The swelling studies of hydrogels wereinvestigated by gravimetric analysis. The prepared samples films were first oven dried till there was no moisture left in them and then were finely powdered with help of mortar-pestle. After powder form was obtained, exactly0.1g of powdered sample was weighed and dipped in 250 ml of swelling medium and allow hydrogel to swell. After this, swollen hydrogels were removed from the swelling medium at regular intervals hydrogels were weighed and placed into the same beakers again. This procedure was carried out as long as a constant weight.The swelling index (SI) was computed using the equation (1) :-

$$SI\,(\%) = \frac{(WS-Wd)}{Wd}\times 100 \qquad (1)$$

Here W_s represents the weight of swollen hydrogel and W_d represents the weight of dried hydrogels.

3.4 Water holding capacity:

In order to calculate the maximum water holding capacity (MWHC) of soil, garden soil was collected and sieved. The sieved soil was then dried until there was no trace of moisture left. In this process four sets with 50g of soil each were prepared. The 3 sets were mixed with 0.1g of hydrogels to make 3 different sets of XG-Fe, NaCMC-Fe, and XG-Fe-NaCMC hydrogels and in one set as a control, a soil sample without hydrogel. All four sets were then placed in a tube (2.5 cm diameter and 9.9 cm height) with small holes at the bottom of tube. The filter paper was placed inside the lower surface and weighed (W_1) to determine the MWHC of the soil. Water was gradually added at regular intervals of two till water begins to leach out of the bottom of the tube. The tube was weighed again once there was no longer any leaking water at the bottom (W_2). The maximum water-holding capacity (MWHC %) of the soil was calculated with the help of equation (2) :-

$$MWHC\% = \frac{(W2-W1)}{50}\times 100 \qquad (2)$$

3.5 Plant growth:

The beneficial effects of the soil amendment using synthesizedXG-Fe-NaCMC hydrogel were observed in plant growth by growing wheat seeds. In this process first garden soil and sieved. The sieved soil was then dried until no amount of moisture was left in the soil. Then four sets of samples were prepared with 300g of soil in each. In each set urea-loaded hydrogel powder (0.1g) of each hydrogels (XG-Fe, NaCMC-Fe and XG-Fe-NaCMC) were placed in soil at a depth of 5cm and one set was taken as a control, in which the soil was treated directly with urea solution. In each set fifteen seeds of wheat were sowed for germination. The experiment was studied for a period of fifteen days, with following actual environmental exposures of each set and all sets receive continuous sunlight and water quantity required for growth from the starting . The germination of seed growth was calculated by counting the number of seeds that sprouted over the period of fifteen days. It was also assumed that the presence of seed radical alone shows the proper sign of germination of seed. After the period of fifteen days the roots and shoots of the germinated plant were carefully separated from the soil and was carefully washed. After the removal of plants from the length of plants were carefully measured from shoots to roots with the help of ruler. Fresh weight of plants were recorded with help of weighing balance and in order to calculate the dry weight of plants the plants were dried at a temperature of 70°C for 24 hours and dried mass of plants were recorded as per accordance.

3.6 Characterization of Hydrogels:

3.6.1 XRD: The X-Ray Diffraction (XRD) analysis of all the three hydrogels (XG-Fe, NaCMC-Fe, XG-Fe-NaCMC) was performed to check the crystallinity of hydrogel by the Model: PerkinElmer spectrum version 10.5.3 in solid-state form.

3.6.2 FTIR Spectroscopy: Fourier transform infrared (FTIR) spectroscopy was performed using Perkin Elmer spectrum two to determine the bonding modes in hydrogels.

3.6.3 Thermo-gravimetric analysis (TGA): TGA of all three hydrogels was carried out using a Perkin Elmer,TGA 4000 by using N_2 atmosphere from 25°C to 800°C with 10°C/min of uniform heating rate to determine the thermal stability of synthesized hydrogel.

3.6.4 Scanning Electron Microscopy: The Surface morphology of all three hydrogels was analyzed by SEM on EVO 18 research, Zeiss, instrument**.**

CHAPTER-4

RESULTS AND DISCUSSION

4.1 Synthesis:

The crosslinking between $FeCl_3$ and mixture of Xanthan gum and NaCMC is pictorially represented in the Fig.5. There were presence of multiple physical cross-linking bond between the Fe^{+3} ion and XG & NaCMC mixture, with presence of hydrogen bonding generated between the chain network of xanthan gum and NaCMC. There is a presence of anionic functional groups in xanthan gum and NaCMC which helps in favoring of ionic crosslinking between the Fe^{+3} ions and the carboxyl functional groups in Xanthan Gum and NaCMC chain network. Because of presence of this type of crosslinking a 3-D network structure is constructed as shown in the fig.5.

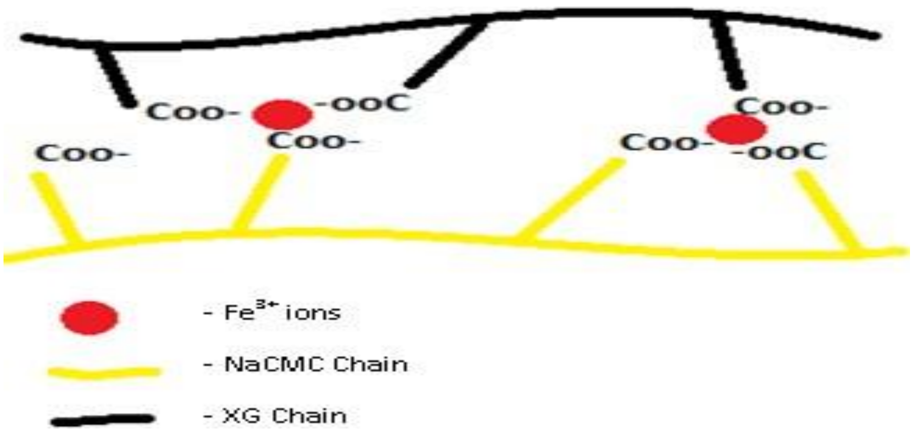


Fig.5. Schematic diagram representation of the preparation of the XG-Fe-NaCMC

4.2 Characterization of synthesized Hydrogels:

4.2.1 X-Ray Diffraction:

X-Ray Diffraction (XRD) pattern of all the three hydrogels (XG-Fe , NaCMC-Fe , XG-Fe-NaCMC) along with XG and NaCMC are plotted and shown in the fig- 6,7 &8 to analyse the morphological nature of hydrogels and compare the crystallinity with there respective precursors from they were synthesized . In the diffractogram of the hydrogels it was seen that there was no trace any sharp peaks in the graph which demonstrates that all the three hydrogels are amorphous in nature and not crystalline. WhereasXG and NaCMC also does not possess any sharp peaks and there is presence of hump in both the case which represents the polysaccharide nature which means xanthan gum and NaCMC are amorphous in nature. XG-Fe and NaCMC-Fe both have a little peak change but XG-Fe-NaCMC is linear. A broad diffraction peak at ≈20° can be seen in XG-Fe which is due to the nature of polysaccharide. However, the intensity of this peak reduced in the mixture hydrogel XG-Fe-NaCMC due to its crosslinking with Fe^{+3} ions.

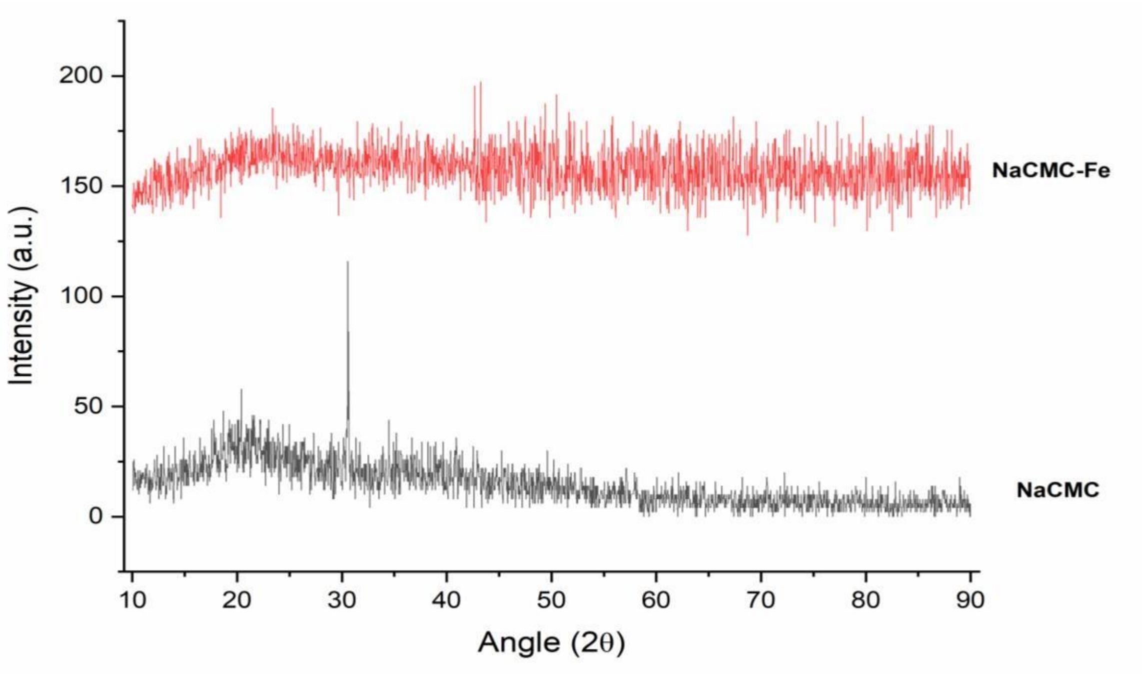


Fig. 6. XRD spectra of NaCMC&NaCMC-Fe

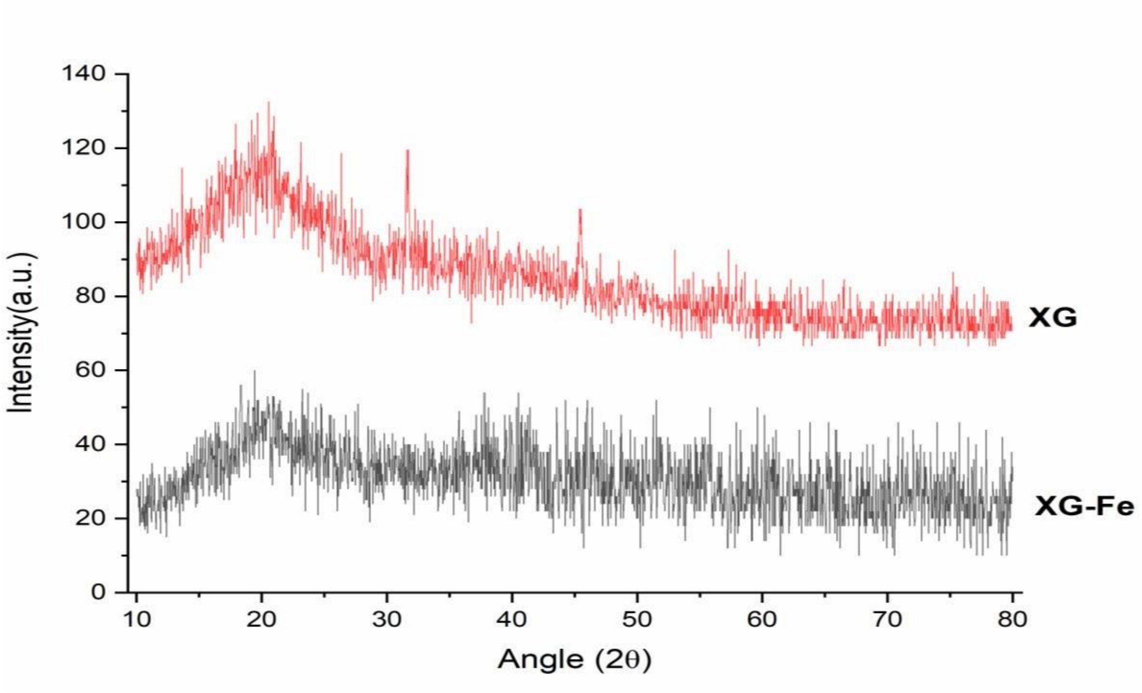


Fig.7. XRD Spectra of XG & XG-Fe

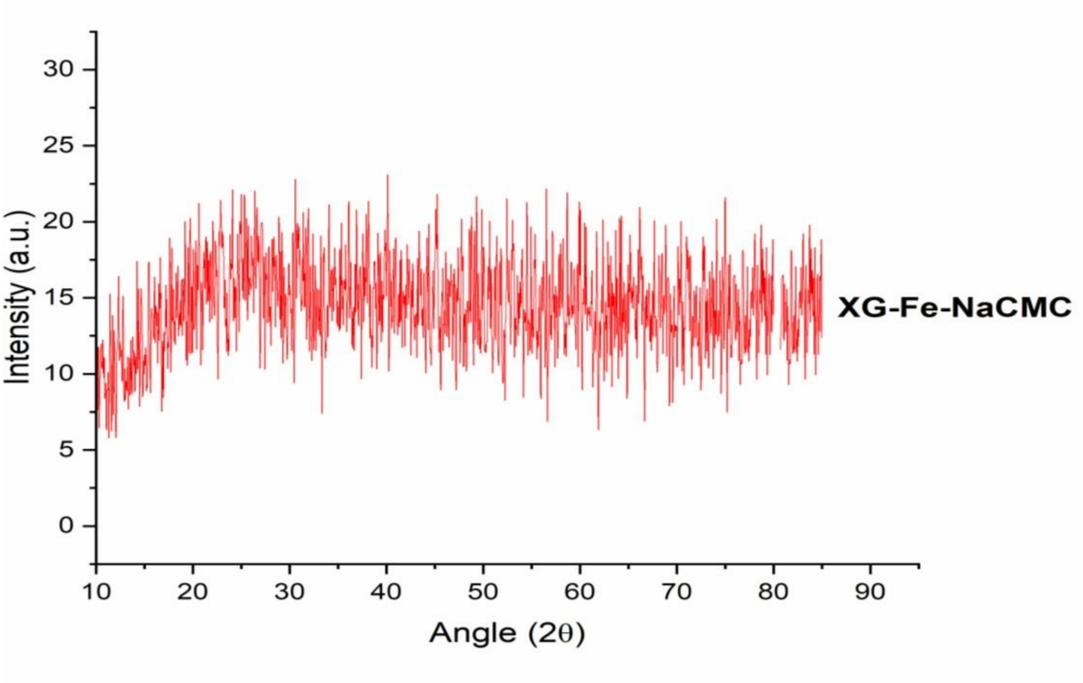


Fig.8. XRD spectra of XG-Fe-NaCMC

4.2.2 Fourier Transform InfraredSpectroscopy:

For the synthesized hydrogels, Fourier transform infrared (FTIR) spectroscopy was performed as shown in Fig 9. The broad peak in the range of 3200-3300 cm^{-1}confirms the presence of-OH groups inthe hydrogel. This intensity is found to be highest in XG-Fe-NaCMC hydrogel. Two broadpeaks between 1609 and 1390 cm^{-1} arise from the asymmetrical and symmetrical stretching mode of $-COO^{-}$ groups. The adsorption band at 2920 and 2850 cm^{-1} correspond to the broadband in region 2980–2800 cm^{-1} of pure XG, which may be assigned to asymmetric and symmetric methylene C-H stretching respectively. IR peaks in the range of 1050-1030 cm^{-1}show the presenceof symmetrical stretching of $-COO^{-}$, agroup of glucuronic acid, and the peak near 1050 cm^{-1} is due to the stretching of C-O-C in the ether group.

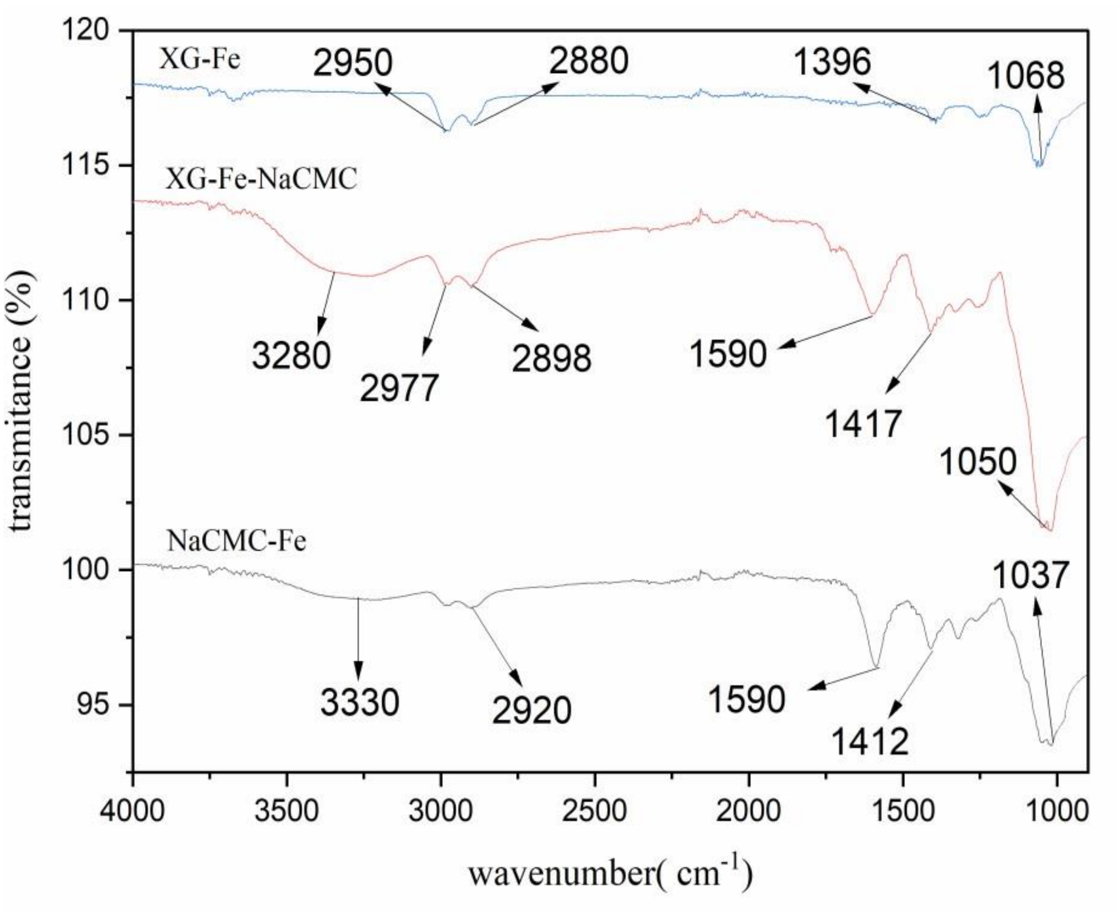


Fig.9. FTIR spectra of hydrogels

4.2.3 Thermogravimetric Analysis:

The display of thermo-gravimetric analysis of all three hydrogels along with Xanthan gum and NaCMC is presented in the fig.10, 11 & 12. In the hydrogels, xanthan gum and NaCMC, it was observed to have a three phase degradation. The first phase was in between the range of 50-200°C which is because of desorption of moisture attributes in hydrogels which is corresponding to the water molecule bounded to the polysaccharide structure. In second phase of degradation a significant amount of weight loss is observed at the temperature range of 250-800 °C. The second phase of degradation is because of breaking down of the main backbone of the polymeric chain network. In sodium salt of carboxymethyl cellulose it can be seen that the second phase of degradation where there is breaking down of polymeric chain begans around 240 °C and there is weight loss from the inorganic portion of chain. In addition, decomposition is caused by the pyrolysis reaction which took place after the temperature reaches above the 335°C. It can be concluded form the TGA data and graph plotted in fig. 12 that the synthesised mixture hydrogel (XG-Fe-NaCMC) has more thermal stability compared with XG-Fe and NaCMC-Fe. Pure NaCMC-Fe hydrogel having lower stability but after addition of XG its stability has increased. Thus, the synthesised hydrogel is thermally stable among others and more efficient alternative to use in place of the pure hydrogels.

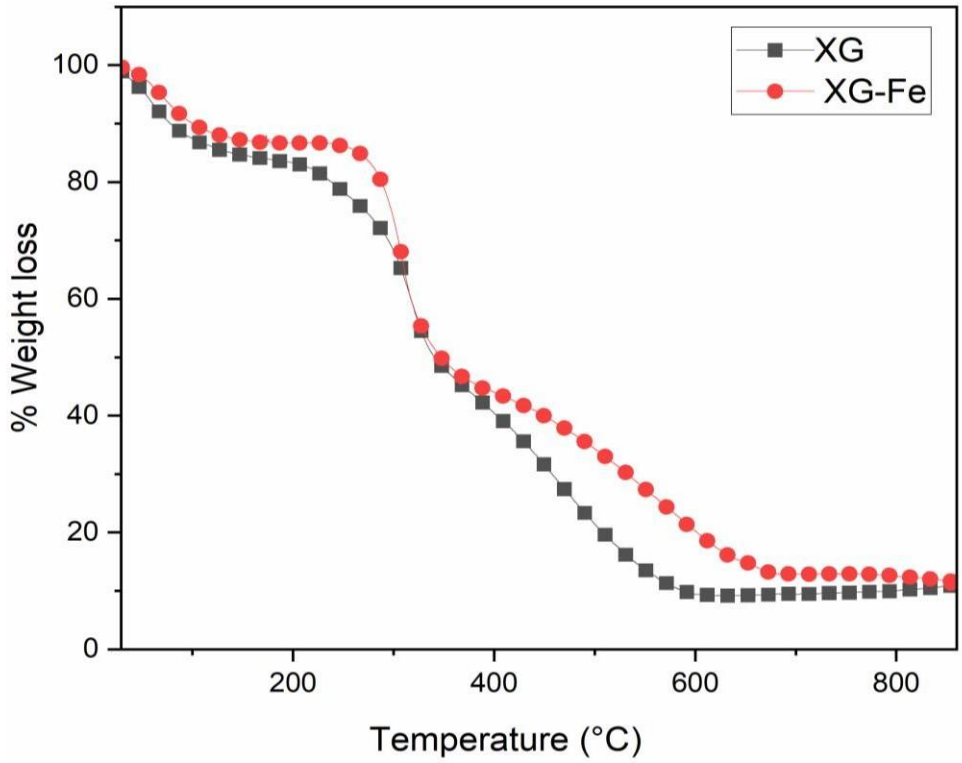


Fig.10 TGA analysis of XG-Fe & XG

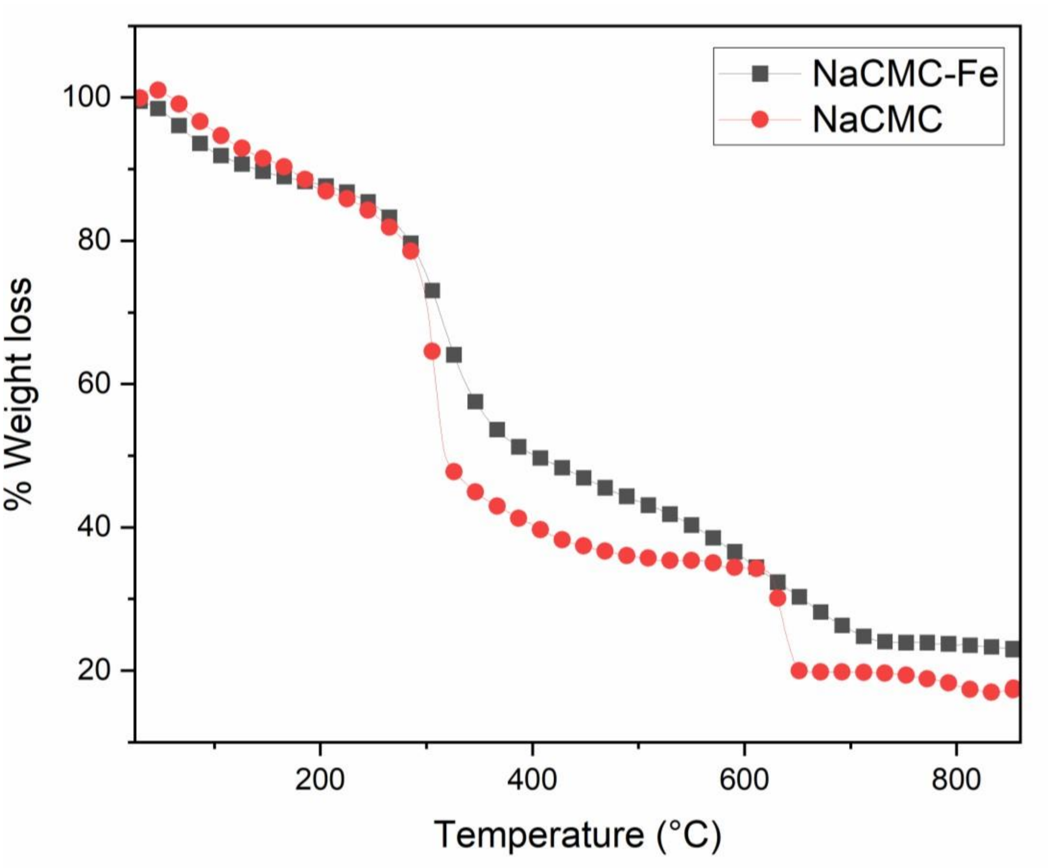


Fig.11. TGA analysis of NaCMC-Fe &NaCMC

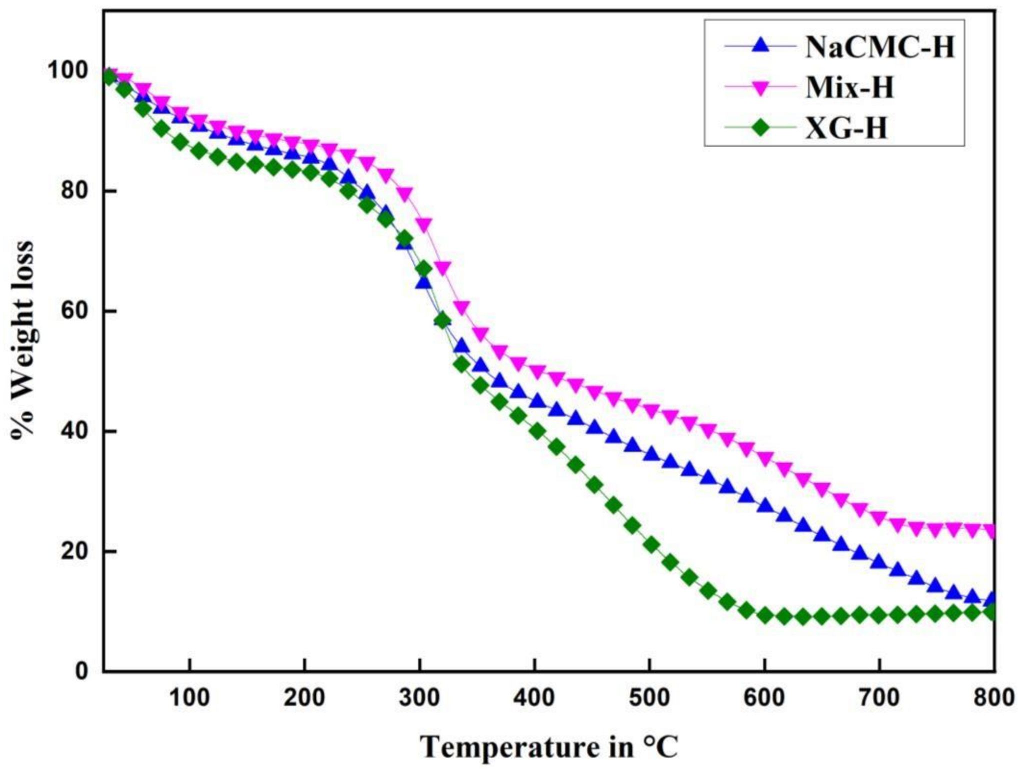


Fig.12. TGA analysis of all three hydrogels

4.2.4 Scanning Electron Microscopy:

Scanning Electron Microscopy (SEM) was performed for the purpose of studying the surface morphologies of all three hydrogels (XG-Fe, NaCMC-Fe, XG-Fe-NaCMC), XG and NaCMC. The results of SEM are as shown in fig. 13 – 17 .It is found that the XG has a plane and smooth surface with less pores on the surface but when the xanthan gum is crosslinked with $FeCl_3$ hydrogel (XG-Fe) has comparatively granular and rough surface with high pores which help in increasing water-holding capacity. The same type of result was present in case of NaCMC, there is also presence of smooth and regular surface but as it is crosslinked with $FeCl_3$ there is a increase in the cracks and holes present on the surface and the surface becomes more rough and highly porous due to presence of these irregularities and cracks on the surface. The SEM images of the hydrogels indicates that the the crosslinking of $FeCl_3$ with \XG and NaCMC generates the high irregularities and smooth surface is converted to rough due to presence of Fe^{+3} ions. However, in the XG-Fe-NaCMC, it was observed to be found most irregular and most porous surface than XG-Fe and NaCMC-Fe. It is because of the presence of two different crosslinked chains present in the mixture which makes its surface highly porous and amorphous, which provides enormous empty space for adsorption of water on the surface of XG-Fe-NaCMC and increase the water holding capacity when mixed with soil to a great extent. The same amorphous nature of the hydrogels is also supported by their XRD data.

EDX was used to check the content of Na and Fe in the Xe-Fe, NaCMC-Fe and XG-Fe-NaCMC . Fe was observed to be 57.56, 54.21, and 87.99 weight % for XG-Fe-NaCMC, NaCMC- Fe, and XG-Fe and hydrogels respectively also the weight % of Na is 24.7 and 31.11 for XG-Fe-NaCMC and NaCMC-Fe.

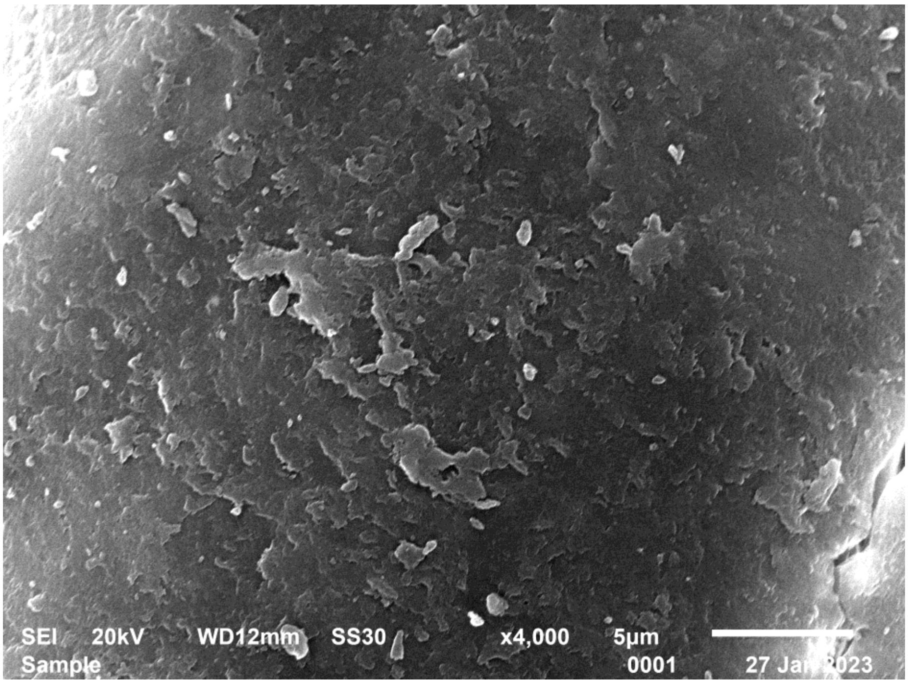


Fig.13. SEM image of XG

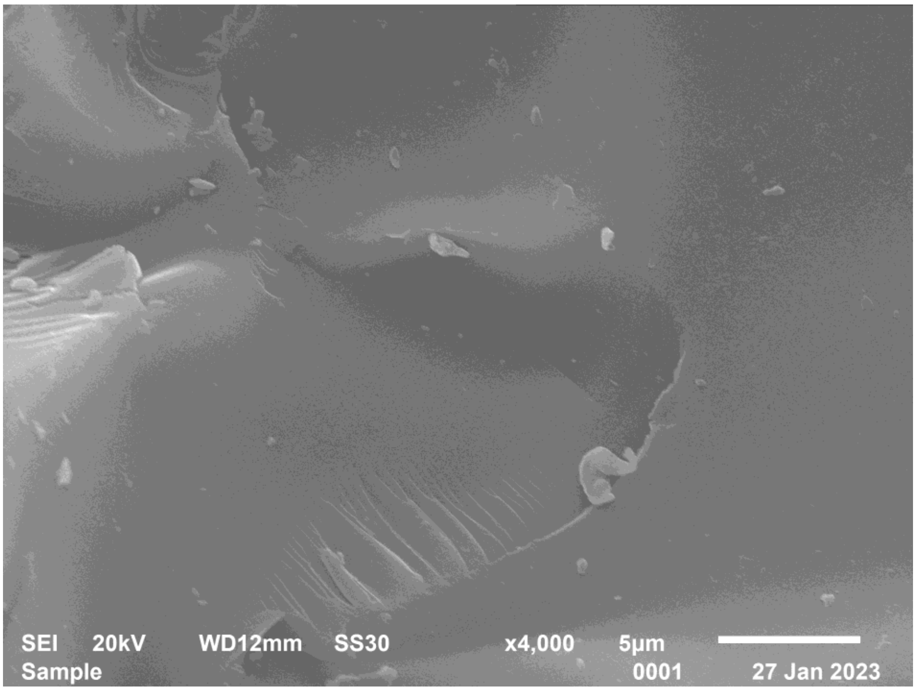


Fig.14. SEM image of NaCMC

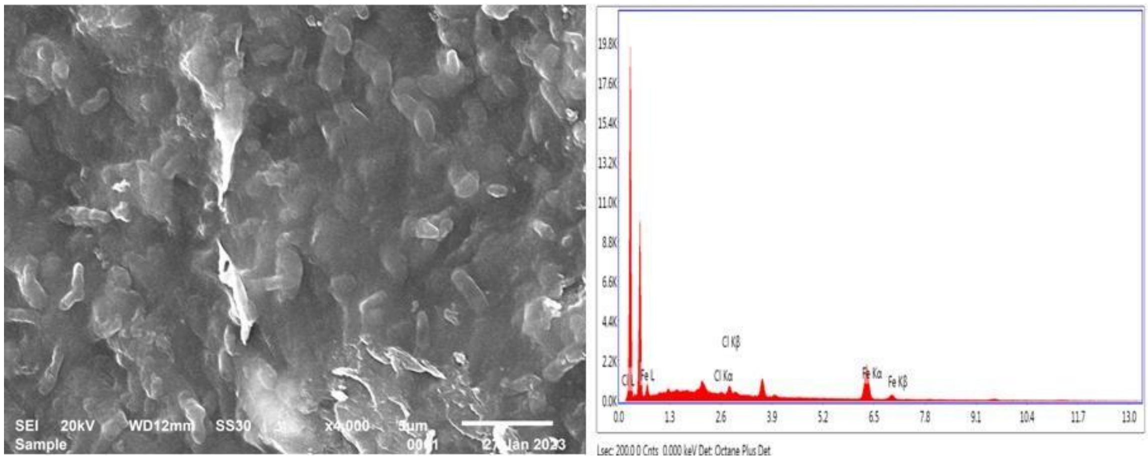


Fig.15. SEM image of XG-Fe withEDX graph of XG-Fe

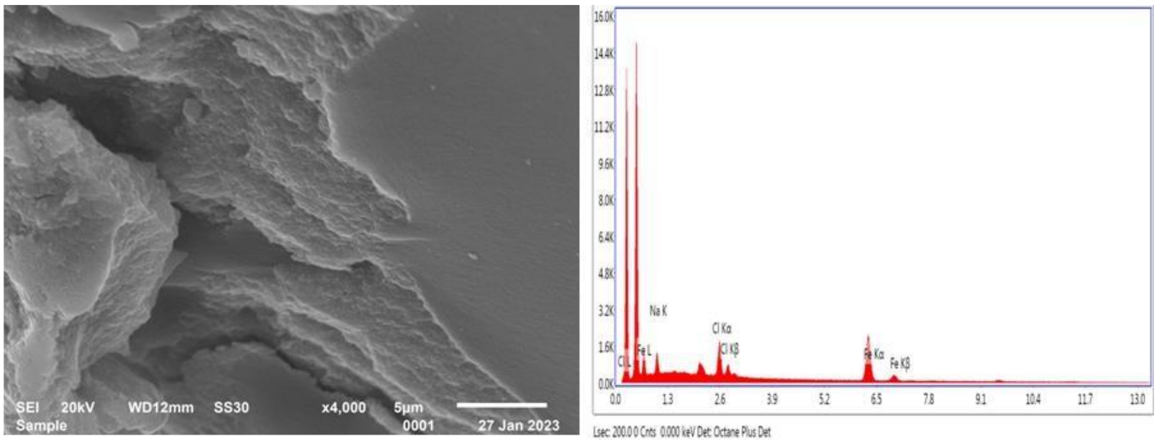


Fig.16. SEM images of NaCMC-Fe with EDX graph of NaCMC-Fe

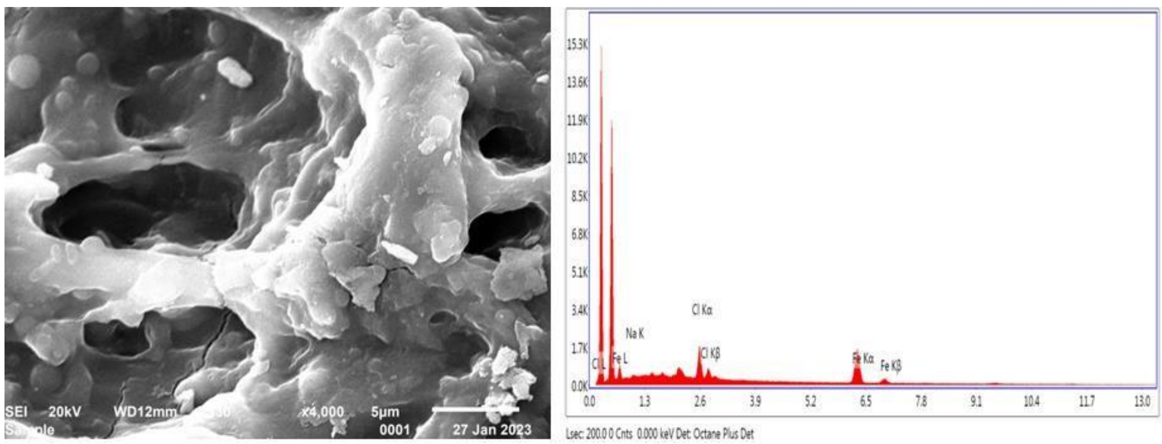
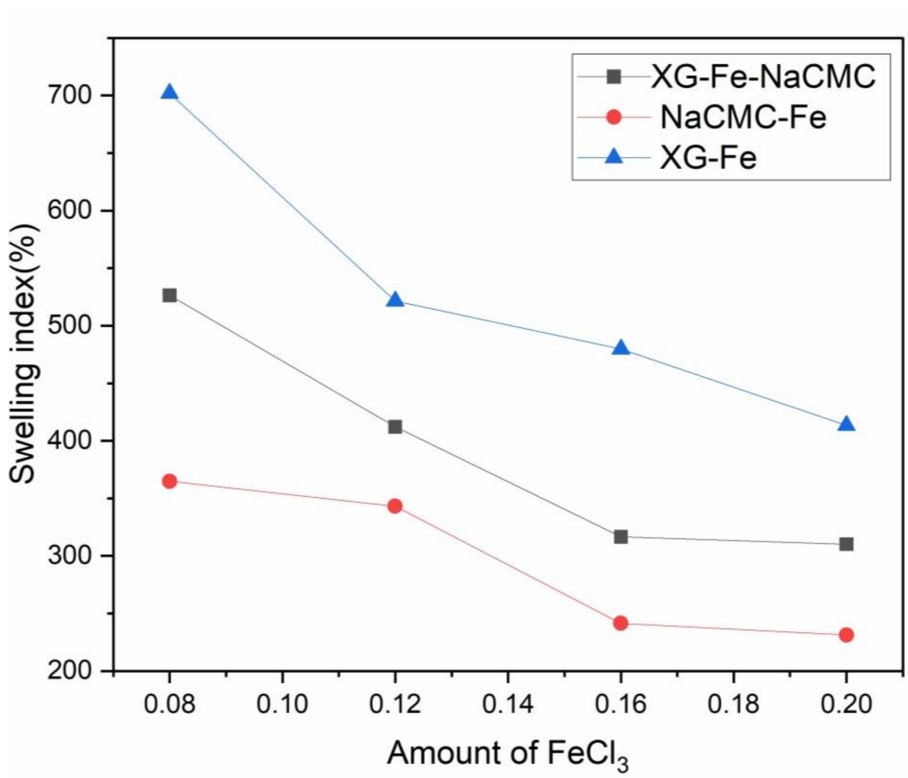


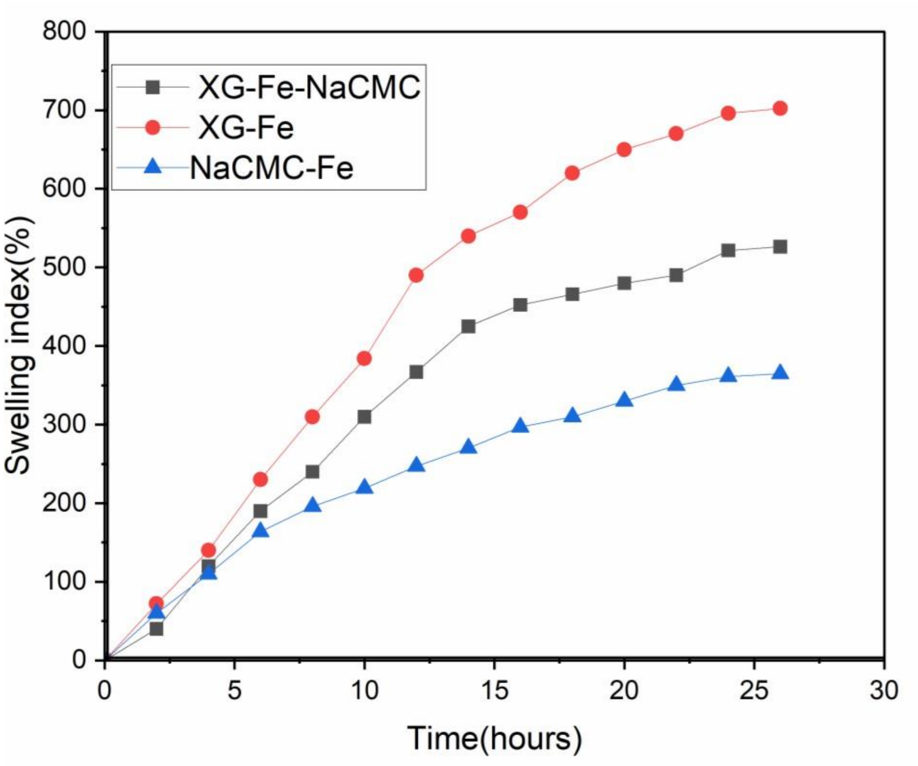
Fig.17. SEM image of XG-Fe-NaCMC with EDX graph of XG-Fe-NaCMC

4.3 Swelling Study:

For the swelling study there has been variation in the amount of crosslinker from 0.04g – 0.2g for the synthesis of hydrogels. From the swelling study performed for the hydrogels, it was clearly shown that there was a decrease in swelling index of all the hydrogels with increasing concentration of crosslinker ($FeCl_3$). It is possible because on increasing the concentration of crosslinker there is a increase in crosslinking network between polymeric chains and Fe^{+3} ions which reduces the empty spaces between the polymeric chain network and thus reduces the void spaces for absorption of water and swell up. Also it was observed that if the concentration of $FeCl_3$ is below 0.08g/40ml the synthesis of hydrogel was not possible due to very low crosslinker concentration. It was noted that swelling index of XG-Fe has a higher value than NaCMC-Fe. On mixing Xanthan Gum with NaCMC and then crosslinking the mixture to synthesize the hydrogel (XG-Fe-NaCMC) there is a increase in the swelling index of the hydrogel when compared to the NaCMC-Fe.



(a)



(b)

Fig.18. (a) Swelling index with respect to amount of $FeCl_3$ (b) Swelling index with respect to time

4.4 Maximum water holding capacity:

The water holding capacity of hydrogels and control has shown in Fig. 19, which was performed for all the three sets of hydrogel (XG-Fe, NaCMC-Fe and XG-Fe-NaCMC) and one without any hydrogel as a reference. In Fig.19, a-soil with Xg-Fe, b- soil with NaCMC-Fe, c- soil with XG-Fe-NaCMC. The result of experiment demonstrates that the control sample i.e. soil without any hydrogel sample had a water holding capacity of 20ml per 50g of soil , whereas soil sample in which NaCMC-Fe was added could hold as much as 22 ml of water. It was also seen that soil sample set in which XG-Fe was added, showed the maximum capacity of water holding out of all four sets and could hold as much as 28 ml of water. Also when XG-Fe-NaCMC sample set was observed and it was found that water holding capacity of soil has increased from 20 ml per 50g soil to 26ml per 50g of soil which is a significant increase of 30% .The results of this experimental study demonstrated that the addition of Xanthan Gum in NaCMC-Fe has increased its water holding capacity to 30% which was before 10% in case of NaCMC-Fe.Hence there is a significant increment in the ability of soil property of water holding and absorbing water when treated with XG-Fe-NaCMC sample [12].

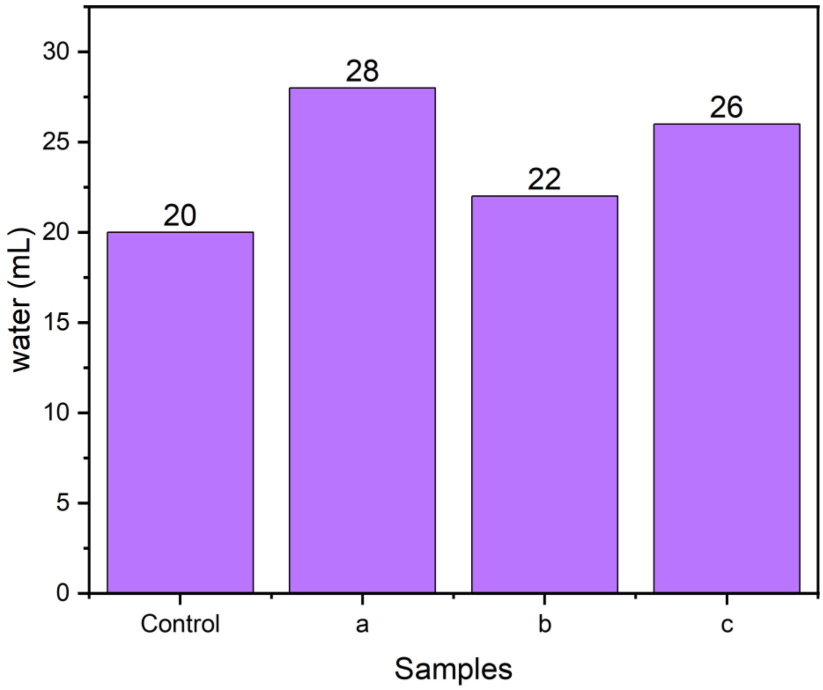


Fig.19, Maximum water holding capacity of hydrogels (a-soil with XG-Fe, b- soil with NaCMC-Fe, c- soil with XG-Fe-NaCMC)

4.5 Plant Growth:

Table-6, displays all the samples data of plant growth that has been germinated for purpose of comparative study of hydrogels on soil and release of agro – chemicals in soil.

The measurement of growth of the wheat grass includes the total change in weight when calculated for total dried sample and fresh set of samples, the average number of seed germinated during the study, the maximum and average height of shoot, the average and maximum length of roots of all the samples.

Table.6. Plants growth measurements

Sample	Total fresh weight of wheatgrass (g)	The total dry weight of wheatgrass (g)	The average number of germinated seeds on the 15th day	Average shoot height (cm)	Average root height (cm)	Growth increase (%)
XG-Fe	2.2696	0.2403	14	18.92	6.48	52
NaCMC-Fe	1.7680	0.1814	14	17.11	5.88	18
XG-Fe-NaCMC	2.6164	0.2745	15	19.14	6.71	75
Ctrl	1.4924	0.1538	13	15.94	5.61	-

With the passage of time there was a regular increase in the plant height which was seen by the naked eyes. At the end 15thday, the sample set with synthesized XG-Fe-NaCMC was recorded to have the tallest plant height with a total increase of 75% more growth than the reference set (set in which urea was directly added). The sample set with XG-Fe was found to have the 2nd best plant growth with a increase of 52% in plant growth which was followed the NaCMC-Fe set which increased the plant growth by a 18%. It can be concluded that the seed growth and germination is better under the condition where there is continous and availability urea for longer time duration.From this comparative study it can be assumed that after combining XG and NaCMC for synthesizing hydrogel, the urea-releasing property got enhanced. Thus, XG-Fe-NaCMC hydrogel is expected to be a potential candidate for the slow-release device of various agrochemicals in agriculture.

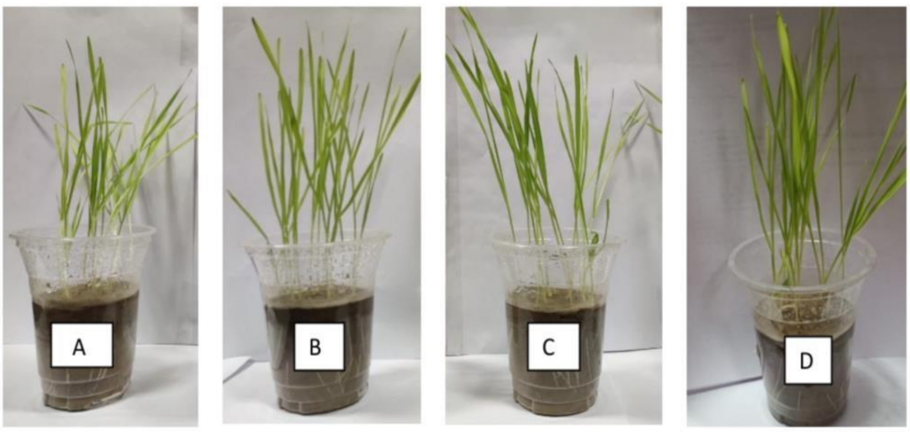


Fig.20, Plant growth, A- Control (Soil without hydrogel), B- soil with NaCMC-Fe, C- soil with XG-Fe, D- Soil with XG-Fe-NaCMC

CHAPTER-5

CONCLUSION AND FUTURE SCOPE

5.1 CONCLUSION: In conclusion, the hydrogel of Xanthan Gum, Carboxymethyl cellulose sodium salt, and their mixture by using ionic crosslinking using (Fe^{3+}) ions are synthesized for agricultural applications. Here, the hydrogel is prepared easily and economically, keeping in view the principles of green chemistry. This is efficient way to use agro waste that is Cellulose and make it useful in the synthesis. The comparative study among three hydrogels revealed the dominance of mixture hydrogel over hydrogels of XG and NaCMC. The mixture hydrogel (XG-Fe-NaCMC) consists of both Fe and Na ions in 57.56 and 24.7 weight %. Also, this mixture hydrogel was found to have better thermal stability and increased plant growth by 75% in terms of total fresh weight comparison with the control. The soil's maximum water-holding capacity was increased by 30% when XG was added with NaCMC for hydrogel synthesis. As a result, the synthesized mixture hydrogel (XG-Fe-NaCMC) has the potential to be employed in agriculture as a water-holding agent as well as a source for Na, and Fe ions.

5.2 FUTURE SCOPE: This Synthesized hydrogel (XG-Fe-NaCMC) is useful in agricultural field. The released study of fertilizers can be studied. As this hydrogel is synthesized by natural sources, it is environment friendly. It is useful for environment friendly manner. This may also act as a carrier for the release of various agrochemicals.

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DEPARTMENT OF CHEMISTRY

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Dear Rahul Yadav, **Dated:22/5/2023**

Thank you for submitting your revised manuscript titled "Synthesis And Characterization Of Xanthan Gum And Carboxymethyl cellulose Sodium Salt-Based Ionic Cross linked Hydrogels For Agricultural Application." We are pleased to inform you that it has been provisionally accepted for publication.

Your manuscript will be given full consideration for inclusion in the special issue of the Bulgarian Chemical Communications to be published during the APM-2023 conference, scheduled for 25-26 April 2023.

We appreciate your contribution and look forward to your participation in APM-2023.

Best regards,

MGH Zaidi

Convener APM-2023

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This is to certify that Mr. Rahul Yadav

from Delhi Technological University, Main

Presented Poster Presentation

In two days national conference, APM-2023 held from 25^{th} to 26^{th} April 2023 at GBPUA&T Pantnagar.

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