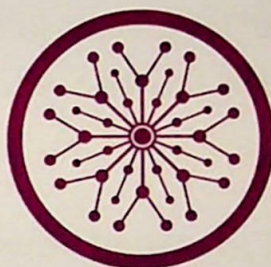


**Synthesis and Characterization of Poly(Gelatin-g-Vinyl Acetate-co-Maleic Acid) and its
Applications**



SUPERIOR UNIVERSITY

Thesis Submitted to

The Superior University Lahore

In Partial Fulfillment of the

Requirement for the Degree of

M.Phil. Chemistry

By

RABBIA SALEEM

SU92-MSCHW-F22-053

Session: 2022-2024

Faculty of Sciences

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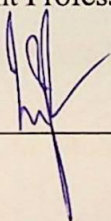
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No part of this thesis has been submitted anywhere else for any other degree. This thesis is submitted to the Faculty of Sciences, The Superior University, Lahore in partial fulfillment of the requirements for the degree of Master of Philosophy in the field of **“Chemistry”** in Faculty of Sciences at The Superior University, Lahore.

Student Name: Rabbia Saleem

Signature: Rabbia

Examination Committee:

Session Chair: Dr. M. Mudassir Iqbal

Signature: [Signature]

a) External Examiner: Dr. Muhammad Aslam

Signature: [Signature]

Associate Professor

Department of Chemistry

University of Education, Lahore

b) Internal Examiner: Dr. Fizza Naseem

Signature: Fizza

c) Supervisor Name: Dr. M. Mudassir Iqbal

Signature: [Signature]

d) Name of HOD: Prof. Dr. Uqba Mehmood

Signature: [Signature]

e) Name of Dean: Prof. Dr. M. Naveed Babur

Signature: [Signature]

f) Controller Examination: Dr. Muhammad Haris

Signature: [Signature]

Dedication

To Rahmat-ul-Lil Aalamin, Hazrat Muhammad (Sallallahu Alaihi Wasallam), whose life and teachings serve as an everlasting source of mercy, inspiration, and guidance for all mankind. This thesis is dedicated to my respected supervisor Dr. M. Mudassir Iqbal, my mentors, whose guidance and wisdom have shaped my academic journey. My family, whose unwavering support and encouragement have been my foundation and my friends, whose companionship and belief in me have been a constant source of motivation.

This thesis is dedicated to all of you.

Acknowledgments

In the name of Allah, the kindest and most sympathetic. All admire Almighty Allah, Who helps and guides us in every darkness of life. Special thanks to Allah Almighty, who showed me courage, patience, and good health, all because I can complete my thesis. All respect and dignity for our Holy Prophet Muhammad (PBUH), Who showed us a light of the right path and provided guidance for us in all aspects of life.

I am extremely thankful to Almighty Allah, who is the source of knowledge and wisdom endowed with mankind, for giving me the acumen and vision to complete this endeavor.

First, I sincerely appreciate my supervisor, **Dr. M. Mudassir Iqbal**, HOD, **Prof. Dr. Uqba Mehmood**, and Dean, **Prof. Dr. M. Naveed Babur**, for their invaluable guidance, encouragement, and constructive feedback. Their expertise and patience have played a crucial role in shaping this thesis. This project would not have been possible without his support and expert guidance.

I would like to extend my deepest gratitude to my family members. Without their encouragement, I would not have been able to complete this endeavor. Ultimately, I am humbly thankful to Almighty Allah, who gave me the courage and abilities to complete this study.

Rabbia Saleem

Table of Contents

	Page
Author's Declaration	II
Plagiarism Undertaking	III
Research Completion Certificate	IV
Certificate of Approval	V
Dedication	VI
Acknowledgements	VII
Table of Contents	VIII
List of Tables	IX
List of Figures	X
List of Abbreviations	XI
ABSTRACT	XII
CHAPTER 1 INTRODUCTION	01
AIMS AND OBJECTIVES	04
CHAPTER 2 LITERATURE REVIEW	05
CHAPTER 3 METHODOLOGY	10
Apparatus	10
Chemicals	10
Synthesis of gelatin grafted copolymer	10
Cross-linking of vinyl acetate with maleic acid	10
Drying of product	11
Characterization	11
Fourier transform infrared spectroscopy (FTIR)	11
Thermogravimetric analysis (TGA)	11
Energy-dispersive x-ray spectroscopy (EDX) coupled with scanning electron microscopy (SEM)	11
Metal and dye absorption	12
CHAPTER 4 RESULTS	13
CHAPTER 5 DISCUSSION	26
CHAPTER 6 CONCLUSION	29
REFERENCES	30

List of Tables

Table No	Description	Page No
3.1	Different ratios of VAc and MA.	10
4.1	Wavenumber with functional group FT-IR Spectra.	13
4.2	Size Calculation for Samples A, B, C and D.	20
4.3	Reaction Kinetics of the removal of metal ions and MB dye.	25
4.4	Reaction Kinetics of the %age removal of metal ions and Mb dye.	25

List of Figures

Figure No	Description	Page No
4.1	Comparative FT-IR spectra of Samples A, B, C, and D.	13
4.2	Thermogravimetric Analysis of Samples A, B, C, and D.	15
4.3	(a) O-K lines, (b) N-K lines, (c) C-K lines for Sample A.	16
4.4	(a) O-K lines, (b) N-K lines, (c) C-K lines for Sample B.	17
4.5	(a) O-K lines, (b) N-K lines, (c) C-K lines for Sample C.	17
4.6	(a) O-K lines, (b) N-K lines, (c) C-K lines for Sample D.	18
4.7	Energy-Dispersive X-ray Spectroscopy (EDX) Analysis result for samples A, B, C and D.	19
4.8	SEM micrographs for samples A, B, C and D.	19
4.9	λ_{max} of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.	20
4.10	λ_{max} of NiCl_2 .	21
4.11	λ_{max} of $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$.	21
4.12	λ_{max} of Methylene Blue.	22
4.13	Calibration curve for $\text{CuSO}_4 \cdot \text{H}_2\text{O}$.	23
4.14	Calibration curve for NiCl_2 .	23
4.15	Calibration curve for $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$.	24
4.16	Calibration curve for Methylene blue.	24

List of Abbreviations

VAc	Vinyl acetate
MA	Maleic Acid
p(Gelatin-g-VA-co-MA)	poly(Gelatin-grafted-Vinyl acetate-co-Maleic acid)
MB	Methylene Blue
AM	Acrylamide
AA	Acrylic Acid
SAP's	Superabsorbent Polymer
EC	Electric Conductivity
IEC	Ion Exchange Capacity
PEG	Polyethylene glycol
DW	Distilled water
DMAAm	N,N-dimethylacrylamide
MBA	N,N'-methylene-bis-acrylamide
TATA	2,4,6-triallyloxy-1,3,5-triazine

ABSTRACT

Recently, hydrogels have been reported to effectively absorb heavy metals from wastewater. The research focuses on developing a novel hydrogel, poly(Gelatin-grafted-vinyl acetate-co-Maleic acid). The poly(Gelatin-g-VAc-co-MA) was synthesized using grafting and co-polymerization techniques. The hydrogel showed effective absorption of heavy metal ions from aqueous solutions.

A free radical polymerization method was used to synthesize poly(Gelatin-g-Vinyl acetate-co-Maleic acid) or [p(Gelatin-g-VAc-co-MA)] with different ratios of monomers, Vinyl acetate (VA) and maleic acid (MA). In this synthesis Maleic acid provides carboxylic group (-COOH) and gelatin provides amine/hydroxyl group (-NH₂/ -OH), facilitating strong binding to heavy metal ions through chelation and ion exchange mechanism.

The FT-IR analysis of the consequent polymer revealed O-H stretching vibrations at 3200-3400 cm⁻¹, C=O stretching of the carboxyl group at about 1720-1740 cm⁻¹ and ester group confirmation peak at 1255 cm⁻¹. Thermogravimetric analysis (TGA) showed that the polymer remained thermally stable up to 300°C, after which it undergoes rapid weight loss between 350°C and 500°C. Energy-Dispersive X-ray spectroscopy (EDX) confirmed the presence of carbon, Oxygen, and Nitrogen in the p(Gelatin-g-VAc-co-MA) polymeric chain. SEM images were taken at a magnification of 30,000x with an accelerating voltage of 10.0kV, displaying a three-dimensional mesoporous structure with a pore diameter of approximately 53.36 nm. The %age removal of metal ions and dye via p(Gelatin-g-VAc-co-MA) proved to be 75.86% for Cu⁺² ions, 84.1% for Ni⁺² ions, 67.1% for Sr⁺² ions, and 74% for methylene blue.

CHAPTER 1

INTRODUCTION

Some of the common contributors of heavy metal pollutants are treated wastewater, industrial effluents, underground water and marine environments [1, 2]. Some of the heavy metals like copper, nickel and zinc are needed by the human body in small quantities but when they are found in large quantities, they become toxic to the living organisms [3]. Metals (copper, strontium and nickel) and dyes may accumulate in body organs, disrupt essential biochemical functions and pose a serious health hazard [4].

The hydrogel and other useful polymeric materials can absorb and retain huge amounts of water, metals, and organic dyes, thus can be used in water treatment applications [5]. Some of the industries that use adsorbent polymers are water-waste treatment [6], hospital bed pads, infant and adult diapers [7], agriculture [8], food processing [9], tissue engineering [10], sensors [11] and medicine delivery just to mention a few [12]

A chemical or physical bond holds the absorbents to create complex three dimensional networks which is a form of molecular web [13]. Due to the chemical or physical cross-linking, these three dimensional polymeric networks are insoluble in water and physiological solutions [14].

Adsorbents are usually polymer chains, cross-linked and thus chemically bonded at different locations passim the polymer backbone [2]. Cross-linking enhances the structural integrity of the polymer when it swells and prevents the polymer to dissolve in water [14].

The polymer chains have hydrophilic (water-attracting) groups such as carboxylate (-COO^-) or hydroxyl (-OH) groups and react with water molecules through hydrogen bonds. The use of these classes is largely due to the ability of adsorbents to absorb metals and dyes [15].

Inside the three-dimensional network, some interrelated pores or voids allow water to participate and be held within the structure [16]. The size and statistical distribution of these pores affects the rate and capacity of water absorption [17].

Vinyl acetate (VAc) is a widely favored material for making absorbents or hydrogels because of its valuable and desirable properties [18]. Due to its properties, it can be used in diverse environments and due to low toxicity, it is safe in highly sensitive environments such as biomedical environments [19]. VAc-based hydrogel may be utilized in tissue engineering or drug delivery applications since it can be prepared to be of different porosity, mechanical strength and swelling profile [20, 21]. Moreover, the compatibility of VAc with other materials allows to create composite hydrogel with better features [22].

Maleic acid (MA) is increasingly utilized in polymer manufacturing because of its low cost [23], non-toxic nature, and good compatibility with a wide range of materials [24].

Through the mechanism of radical recombination that initiates the basic reaction of maleic acid, the use of maleic acid to react with VAc can offer further functionalities to the polymeric substrate through chemical reactions [25]. It is possible because maleic acid ($C_4H_4O_4$) can combine with the vinyl groups of VAc because it is a dicarboxylic acid and has two carboxylic groups [26].

This reaction can modify the structure of the polymer by generating the radical species of both the vinyl acetate and the maleic acid [25]. Depending on the procedure, this process can change the mechanical strength of the polymer, its cross-linking density, or reaction to external stimuli among other valuable features [25].

The hydrophilic monomers are used in the preparation of temperature-sensitive hydrogels together with N, N-isopropylacrylamide that has a hydrophobic counterpart, isopropyl [27]. On the contrary, chitosan, cellulose, gelatin, poloxamers, and their derivatives are excellent starting materials in the production of thermoresponsive hydrogel [28]. Conversely, hydrogels that are pH-sensitive have to be formed by incorporating cationic and anionic functional groups. This addition ensures the responding and adjustment of the hydrogel to the different environmental acidity or alkalinity conditions [29]. Hydrogels responding to the pH are produced by responding natural polymers of albumin, gelatin, alginate, chitosan and carboxymethyl dextran, with acrylic acid derivatives [30]. Thus, the creation of smart hydrogels is linked to the necessity to find new monomer structures that are pH- and temperature-sensitive.

These materials are highly advanced and have numerous applications in practice and adapt to the surrounding [31].

The rise of interest has necessitated the establishment of new metal and dye adsorbents using the advantageous and affordable compounds [32]. Maleic acid and VAc are offered as a potential opportunity to utilize the beneficial characteristics of the substance and meet the requirement of a high-quality and low-cost material since a new type of a metal and dye adsorbent can be developed [33].

AIMS AND OBJECTIVES

Objectives of this study are:

- Synthesis of poly(Gelatin-g-VAc-co-MA)
- Characterization of poly(Gelatin-g-VAc-co-MA)
- Evaluation of metal ion absorption by poly(Gelatin-g-VAc-co-MA)
- Evaluation of organic dye absorption by of poly(Gelatin-g-VAc-co-MA)

CHAPTER 2

LITERATURE REVIEW

Heavy metal ions are among the most hazardous water-soluble pollutants, as they readily dissolve in water and pose serious risks to both human health and the environment [1]. Even low levels of these metals can bring about numerous health problems including weight loss, hearing loss, diarrhea, muscle weakness, stunted growth, cardiovascular problems, cancer, kidney failure and disturbed sleep [34].

Development of the earliest superabsorbent polymer was the starting point in the decades long development of adsorbent materials. The heating and polymerization of the divinylbenzene and acrylic acid (AA) in water was what made this innovation possible as a network was created which could absorb and hold much liquid. This innovative achievement gave way to subsequent achievements in the sphere of polymers that absorb water [35].

Hydrolysates of starch graft acrylonitrile (HSPAN) were first developed in horticulture and agriculture by scientists in the Northern Regional Research Lab of the USDA. These hydro gels were designed to ensure good growth of plants as they enhance the ability of the soil to retain water [36].

Acrylonitrile was grafted onto wheat starch, creating a superabsorbent product with the remarkable ability to absorb 300-1,000 times its water weight and excellent water retention properties. Although the product offered many benefits, it struggled with high production costs and certain structural limitations. These issues initially held back their success in the market [37].

The first commercial adsorbent was produced through the alkaline hydrolysis of starch-grafted polyacrylonitrile. This achievement represented a significant advancement towards the large-scale production of adsorbents (SAPs), facilitating their widespread industrial application in various fields [36].

Adsorbents (SAP's) were mass-produced and quickly became popular for hygienic applications, first gaining widespread use in the United States and Japan. Their remarkable ability to absorb and retain moisture completely changed the industry,

greatly improving the effectiveness and comfort of products like diapers and sanitary napkins [38].

Different polyvinyl acetate ratios were mixed with acrylamide and ferric chloride nanoparticles and exposed to varying doses of gamma irradiation. This process helped form a three-dimensional network, enhancing its ability to clean contaminated water [39].

Vinyl acetate and acrylic acid were covalently attached to a gelatin backbone with ammonium persulfate as an initiator in order to produce a novel biodegradable copolymer and nanocomposite of the polymer, which contained nickel-doped zinc oxide nanoparticles. These materials were prepared by the free radical polymerization process and detailed. FTIR analysis confirmed the successful attachment of monomers to the gelatin backbone [40]. According to TGA results, the copolymers remained stable when heated up to 500 °C. The incorporation of nanoparticles further enhanced their thermal stability, making them more resistant to degradation at elevated temperatures [41]. As per degradation studies, the copolymer had a weight loss of 25.29 percent in two months, and 15.01 percent in the nanocomposite. This implies that durability and biodegradability is enhanced by addition of nanoparticles. In soil burial, the materials initially showed that they had adsorbed water and thereafter weight loss [42].

There are many hydrophilic copolymers that are utilized in pharmaceutical and biomedical sectors among other chemical industry sectors. As an example, the graft copolymers utilized in tablet coating include poly(ethylene glycol)-poly(vinyl alcohol) in offering controlled release of active ingredients [43]. These copolymer grafts are expressed on polyethylene glycol to generate vinyl acetate grafts on polyethylene glycol and subsequently hydrolyzed to produce poly(ethylene glycol)-poly(vinyl acetate) grafts copolymers. Such materials vary in molar mass and chemical composition and are often polyethylene glycol and polyvinyl acetate homopolymers [44].

It is a biodegradable food packaging material, an antibacterial biodegradable starch-based composite, which is more so eco-friendly. Vinyl acetate was graft polymerized with native potato starch under alkaline condition by using ammonium persulfate at 70 to 80 °C [45]. The grafting process showed high effectiveness, achieving 82%

efficiency and a 91.5% conversion rate of vinyl acetate. By incorporating polyhexamethylene guanidine chloride (PHMG-ch), which possesses strong antibacterial properties, a biodegradable composite was obtained. The starch-g-PVAc + PHMG-ch material demonstrates its sustainability through potential applications in water treatment, medical use, and food packaging [46].

The poly(acrylic acid-co-acrylamide-co-maleic acid) [p(AA-co-AM-co-MA)] adsorbent was synthesized via free radical copolymerization of acrylic acid (AA), acrylamide (AM), and maleic acid (MA). Maleic acid played a key role in enhancing the performance of the polymer. Under optimal conditions, the adsorbent exhibited exceptional water absorption capacity, reaching 1348 g/g in distilled water and 106 g/g in a 1.0 wt% NaCl solution. The polymer is also significantly effective in pseudo glucose and urea solutions. This is due to its smart, adaptive behavior as it responds to changes in the environment like temperature, pH and the level of salt, which makes it useful in a variety of applications, including improved control of water adsorption and retention [47].

In order to minimize water contamination by agricultural fertilizers, high swelling cross-linked hydrogel were produced using maleic acid (MA) and N,N-dimethylacrylate (DMAAm) by utilizing redox polymerization. The swelling properties along with the urea sorption/release kinetics of these hydrogels in deionized water at diverse conditions were identified. They were named DM1, DM2 (with the addition of HCl) and DM3 (with the addition of NaOH). The Langmuir isotherm was used to bring out effective urea uptake weights as it provided the best fit to sorption studies. The release behavior of urea was evaluated using mathematical models such as zero-order, first-order, Higuchi, and Korsmeyer-Peppas, which reflected the pH-responsive and functionally modified characteristics of the hydrogel. DM1, DM2 and DM3 liberated 100 percent of their urea in 48, 41 and 18 hours respectively at pH 10, pH 8, and pH 8 respectively. These findings point out the ability of the hydrogels to be effective as urea fertilizer carriers, which have control of releasing functionalities that maintain low environmental pollution and advance agricultural sustainability [47].

Super-swelling acrylamide (AAm)/maleic acid (MA) hydrogels were synthesized through free radical polymerization in aqueous medium, using MA as a comonomer

and multifunctional crosslinkers such as 1,4-butanediol dimethacrylate and trimethylolpropane triacrylate. Their adsorption and swelling behavior were investigated using Basic Blue 17 (Toluidine Blue), a water-soluble monovalent cationic dye. Dynamic swelling studies revealed that the proportion of MA strongly affected the swelling properties of the hydrogels. While AAm-only hydrogels exhibited swelling capacities between 780% and 1360%, the AAm/MA hydrogels demonstrated remarkably higher values, ranging from 1660% to 6050%. The equilibrium water content of AAm/MA hydrogels was also significant, lying between 0.8873 and 0.9837. Water uptake followed a non-Fickian diffusion mechanism. Adsorption experiments performed at 25 °C using a batch method indicated that dye uptake conformed to the S-type isotherm (Giles classification). Increasing the MA content in the hydrogels enhanced the binding ratio with the dye, thereby improving overall adsorption efficiency [48].

A detailed investigation was carried out on the synthesis and characterization of eco-friendly absorbent polymers derived from activated charcoal. In this process, glycerol and activated charcoal underwent polymerization initiated by sodium hydroxide, with maleic acid serving as the crosslinking agent. The resulting products were designated as HCG-1 and HCG-2 hydrogels. These hydrogels were characterized using techniques such as scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared (FTIR) spectroscopy. During optimization, the concentrations of maleic acid and activated charcoal were varied, along with adjustments in swelling time. FT-IR analysis showed the presence of strong, sharp peaks in HCG-1 at 1591.34 and 1400.28 cm^{-1} . These were asymmetric bending vibrations and $-\text{COO}^-$ symmetric stretching vibrations implying the reacting monomers had interlinked successfully [49].

Poly(acrylamide-maleic acid) hydrogel was synthesized by free radical polymerization of acrylamide and maleic acid, employing N,N'-methylene bisacrylamide as a cross-linker and potassium persulfate as an initiator. The obtained hydrogel was characterized using Fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscopy (FE-SEM), thermogravimetric analysis (TGA), and UV-visible spectroscopy. Its adsorption ability was evaluated for the removal of malachite green (MG), a cationic dye, from aqueous solution. The hydrogel demonstrated outstanding removal efficiency with very high adsorption

capacity, reaching equilibrium within 60 minutes and following pseudo-second-order kinetics [50].

Further studies investigated the influence of different cross-linking agents, including N,N'-methylene bisacrylamide (MBA) and 2,4,6-triallyloxy-1,3,5-triazine (TATA), on the swelling and de-swelling behavior of the hydrogels at varying maleic acid contents. The effects of cross-linker and initiator concentrations on swelling were systematically analyzed, along with the impact of pH changes and salt solutions simulating biological fluids [51].

CHAPTER 3

METHODOLOGY

3.2 Chemicals

Gelatin, cetyltrimethylammonium bromide (CTAB), vinyl acetate, maleic acid, sodium hydroxide (NaOH), potassium per sulphate (KPS), N, N-methylene bisacrylamide (MBA), ethanol, buffer solution of pH 4, methylene blue, heavy metal salts, and distilled water. Every chemical and reagent utilized in the studies was of analytical grade.

3.3 Synthesis of Gelatin Grafted Copolymer

In a two-neck reaction flask fitted with a mechanical stirrer, 2.5 g of gelatin was dissolved in 20 mL of double-distilled water by heating the mixture to 70 °C until a clear solution was obtained. Following this, CTAB (0.5 g) was introduced as an emulsifier to ensure proper dispersion and stabilization of the solution [47].

3.4 Cross-Linking of Vinyl Acetate with Maleic Acid

The smart metal and dye adsorbent p(Gelatin-g-VAc-co-MA) was synthesized by dissolving 4.37 g of vinyl acetate (VAc) monomer and 1.47 g of maleic acid (MA) in 20 mL of distilled water under magnetic stirring. The pre-formed gelatin-grafted copolymer was then added gradually to this solution, followed by the addition of 2.00 g of NaOH and 0.0045 g of MBA as the cross-linker. The reaction mixture was heated above 60 °C, after which 0.018 g of KPS was introduced as the initiator, and the polymerization was continued for 2 hours [52].

All optimization reactions were carried out in the same way with different quantities of MA and VAc, resulting in four absorbent samples as listed in Table 3.1.

Table 3.1 Different ratios of VAc and MA.

Name	VAc	MA
Sample A	90%	10%
Sample B	80%	20%
Sample C	70%	30%
Sample D	60%	40%

3.5 Drying of Product

The synthesized adsorbent was initially dried at room temperature, followed by immersion in distilled water to eliminate homopolymers and unreacted residues. Finally, it was oven-dried at 60 °C [47].

3.6 Characterization

Structural and morphological properties of poly(Gelatin-g-VAc-co-MA) were analyzed using Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA), Energy-Dispersive X-ray Spectroscopy (EDX), and Scanning Electron Microscopy (SEM). A brief description of each technique is as follows:

3.6.1 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR was employed to investigate the chemical structure of the adsorbent polymer. The dried material was finely powdered, mixed with KBr, and compressed into pellets for measurement. Spectra were recorded in the range of 4000-400 cm^{-1} using an FTIR spectrometer [53, 54].

3.6.2 Thermogravimetric Analysis (TGA)

TGA was conducted to assess the thermal stability of the polymer by monitoring weight changes as a function of increasing temperature. These weight changes can result from chemical processes such as decomposition and oxidation, as well as physical phenomena like sublimation, vaporization, and desorption [55].

In TGA, the polymer was initially dried in a vacuum oven to fix the weight, and then the adsorbent polymer was heated gradually, temperature range from 30°C- 900°C, and the weight loss of the adsorbent polymer was recorded.

3.6.3 Energy-Dispersive X-ray Spectroscopy (EDX) Coupled with Scanning Electron Microscopy (SEM)

EDX is an analytical technique employed to determine the elemental composition and chemical characteristics of polymers. When combined with SEM, it enables simultaneous evaluation of surface morphology and spatially resolved elemental distribution, providing comprehensive insights into the structure of the material. High-energy electrons 10.0kV are allowed to fall over the surface of the adsorbent

polymer, characteristic x-rays are emitted as secondary back-scattered electrons that are detected as high-resolution, 3D images with magnification up to 30,000x [56].

3.6.4 Metal and Dye Absorption

Poly(Gelatin-g-Vinyl acetate-co-Maleic acid) was treated with copper sulphate pentahydrate, Nickel chloride, Strontium chloride hexahydrate, and methylene blue along with a buffer solution of pH-4 [57].

The absorbance of aqueous solutions containing metal ions and dye was measured using a single-beam Spectrophotometer after determining the λ_{max} of each solution, within the wavelength range of 400-800 nm.

Calibration curves were prepared with five standard concentrations of each metal ion and methylene blue dye, plotting concentration on the x-axis against absorbance on the y-axis. Based on Beer-Lambert's law, the concentration of the unknown solution was calculated, and the percentage removal of metal ions and dye was determined using the respective formula [58]:

$$\% \text{age metal ion/MB removal} = \frac{[C_0 - C_t]}{C_0} \times 100 \dots\dots\dots 1$$

Where:

C_0 is the concentration of metal ions/MB dye before treatment.

C_t is the concentration of metal ions/MB dye after treatment.

CHAPTER 4

RESULTS

4.1 Structural Techniques

4.1.1 Fourier Transform Infrared Spectroscopy (FT-IR)

FTIR analysis was carried out to identify the functional groups present in the polymer backbone.

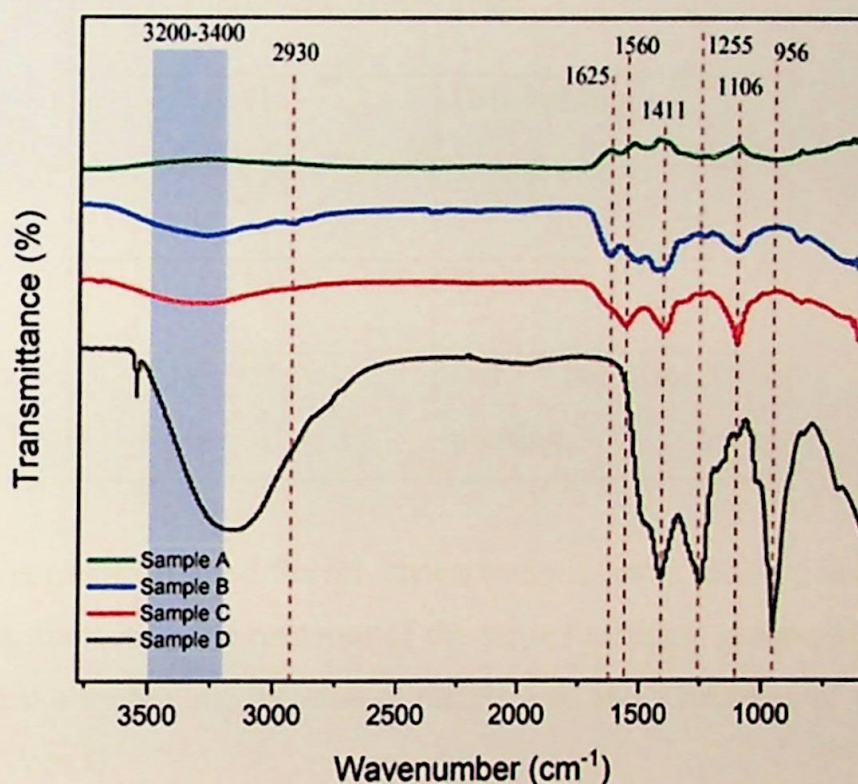


Fig. 4.1 Comparative FT-IR spectra of Samples A, B, C, and D.

The spectra are plotted as transmittance against wavenumber (cm^{-1}), with characteristic peak positions clearly indicated. This FTIR spectrum corresponds to Sample A, B, C, and D IR Spectra, recorded using a Thermo-Fisher Scientific instrument.

Fig. 4.1 shows that O-H stretching vibrations are responsible for the broad and powerful absorption peak located at 3200-3400 cm^{-1} . The absorption band observed

at 1720-1740 cm^{-1} corresponds to the C=O stretching vibration of the carboxyl group. Peaks around 2930 cm^{-1} are attributed to C-H stretching, while the band near 1106 cm^{-1} is associated with C-O stretching.

Table 4.1 Wavenumber with functional group FT-IR Spectra.

Wavelength (cm^{-1}) ¹	Functional group
3200-3400	O-H Stretching
2930	C-H Stretching
1720-1740	C=O
1560-1625	C=C
1411	C-H bending
1255	C-O-C
1106	C-O
956	Out of the plane C-H bending

The spectra correspond to different concentrations and exhibit peaks at similar wavenumbers, indicating the presence of the same functional groups. However, this FT-IR confirms a systematic increase of maleic acid and a decrease of vinyl acetate from Sample A to D.

This compositional change is clearly reflected in the FT-IR spectral changes, where increased maleic acid leads to more pronounced signals associated with carboxylic acid groups, while decreasing vinyl acetate results in less prominent ester-related peaks. Sample-D, having the highest maleic acid content, exhibits the richest chemical functionality among all samples.

4.2.1 Thermogravimetric Analysis (TGA)

The TGA results indicate that all samples A, B, C, and D remain thermally stable up to 300°C, after which they undergo rapid weight loss between 350°C and 500°C.

Variations in the residual weight at higher temperatures are likely due to differences in composition, with Sample D having higher concentrations of maleic acid begins to lose weight at a lower temperature and drops more steeply than other samples, suggesting it is less thermally stable.

Overall, the thermal decomposition patterns are quite similar across the samples, suggesting that samples with high maleic acid decompose at lower temperatures, and increased maleic acid content reduces thermal stability.

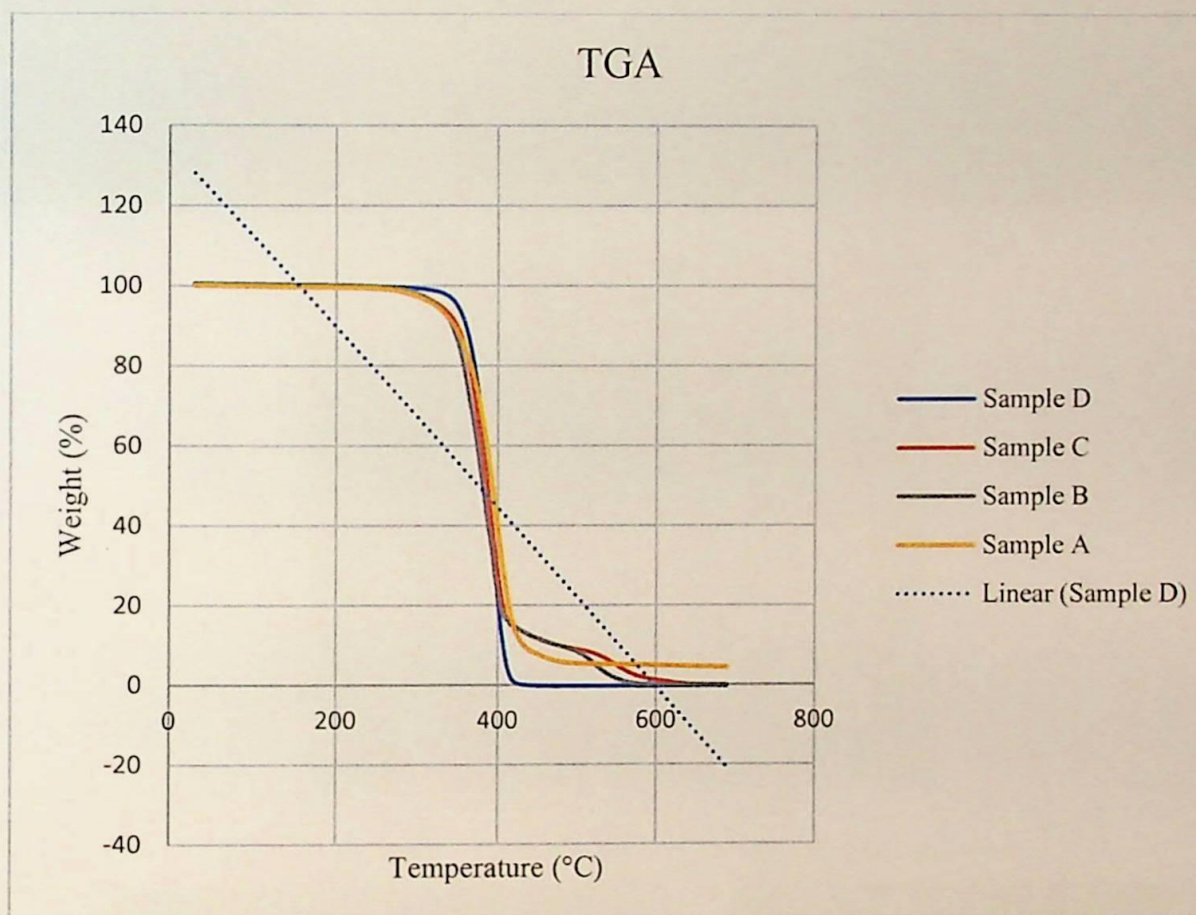


Fig. 4.2 Thermogravimetric Analysis of Samples A, B, C, and D.

4.3 Energy-Dispersive X-ray Spectroscopy (EDX) Coupled with Scanning Electron Microscope (SEM)

Energy-Dispersive X-ray Spectroscopy (EDX), in combination with Scanning Electron Microscopy (SEM), revealed the elemental composition of the samples. The spectra displayed characteristic K-shell X-ray emission lines corresponding to oxygen (O-K), nitrogen (N-K), and carbon (C-K). These signals confirm the

presence and distribution of these elements within samples A, B, C, and D, thereby providing insights into their chemical composition and surface characteristics.

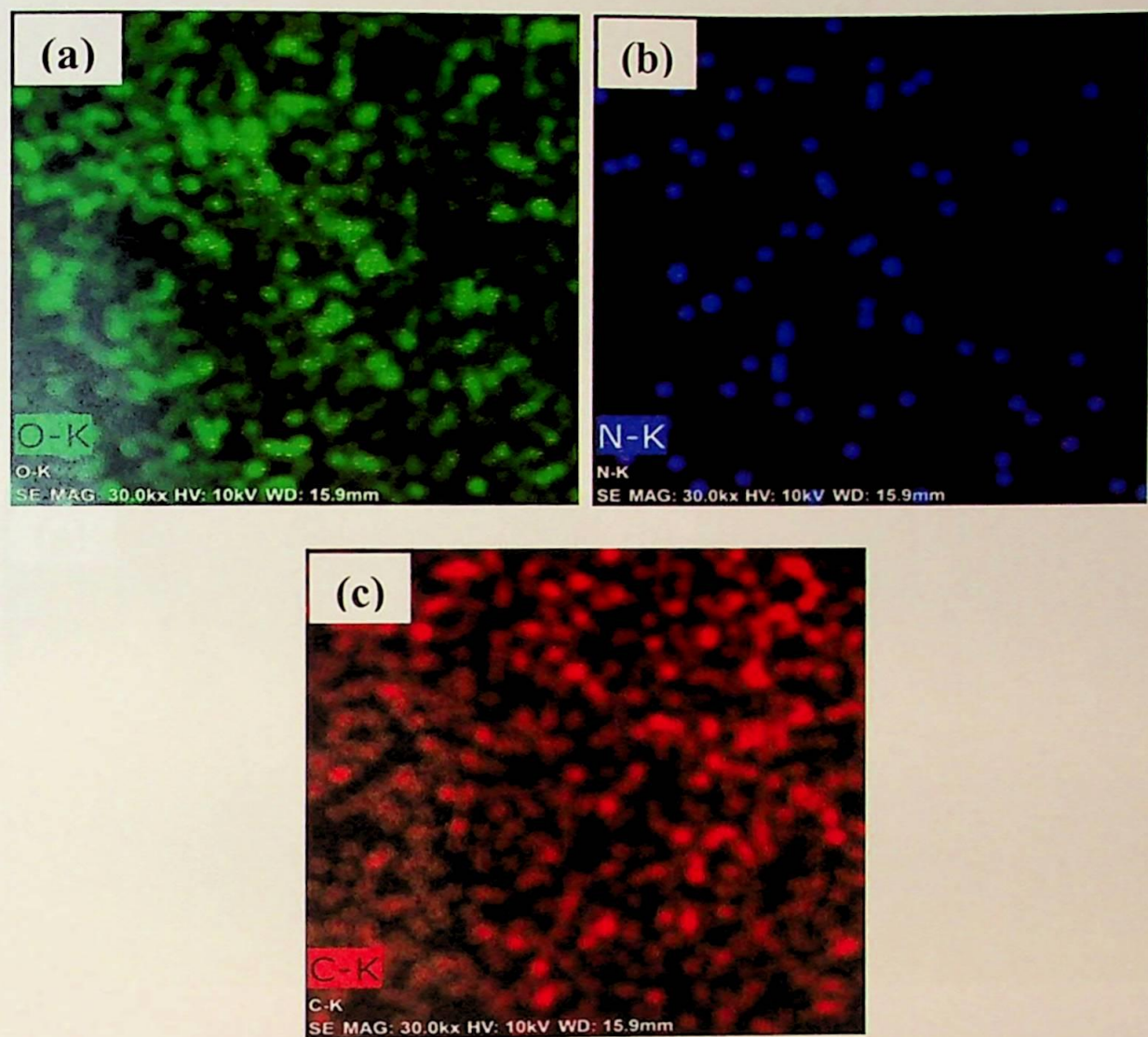
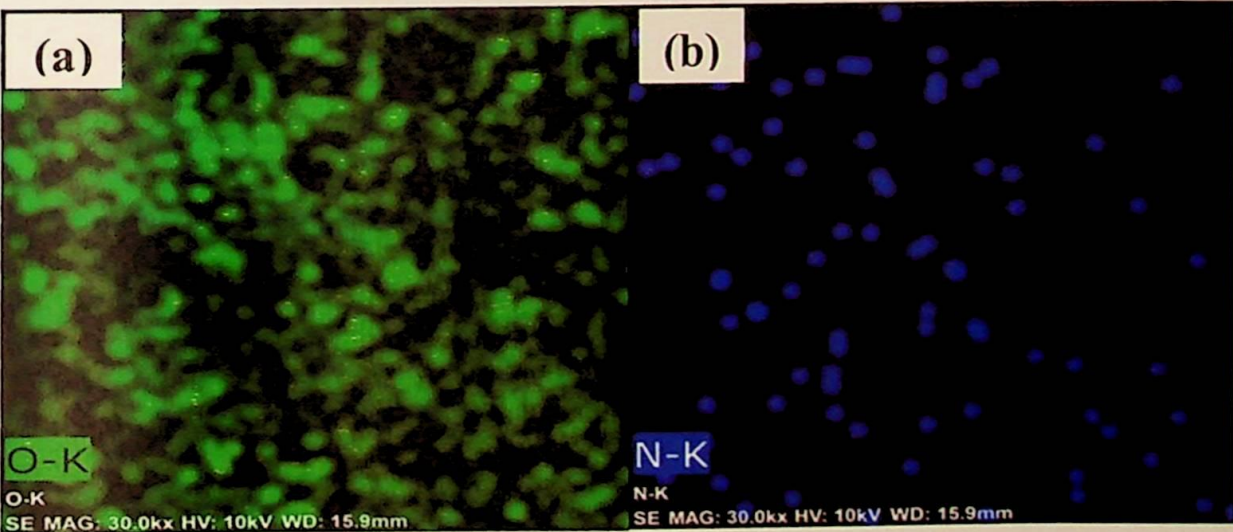


Fig. 4.3 (a) O-K lines, (b) N-K lines, (c) C-K lines for Sample A.



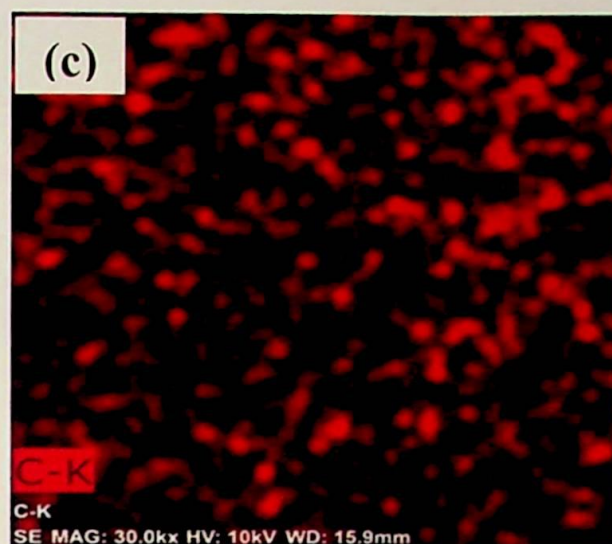


Fig. 4.4 (a) O-K lines, (b) N-K lines, (c) C-K lines for Sample B.

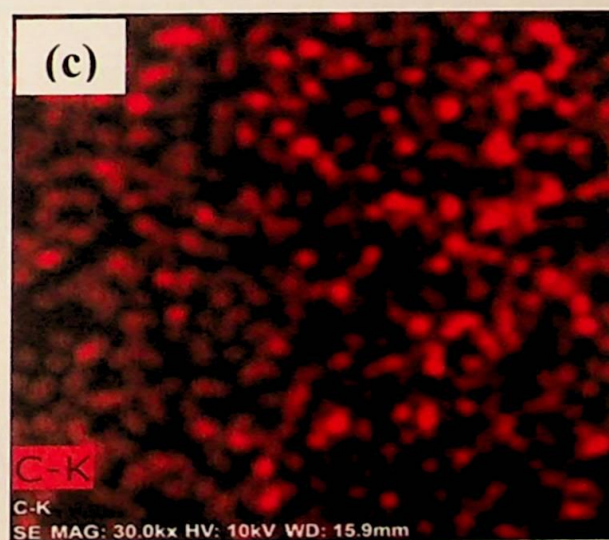
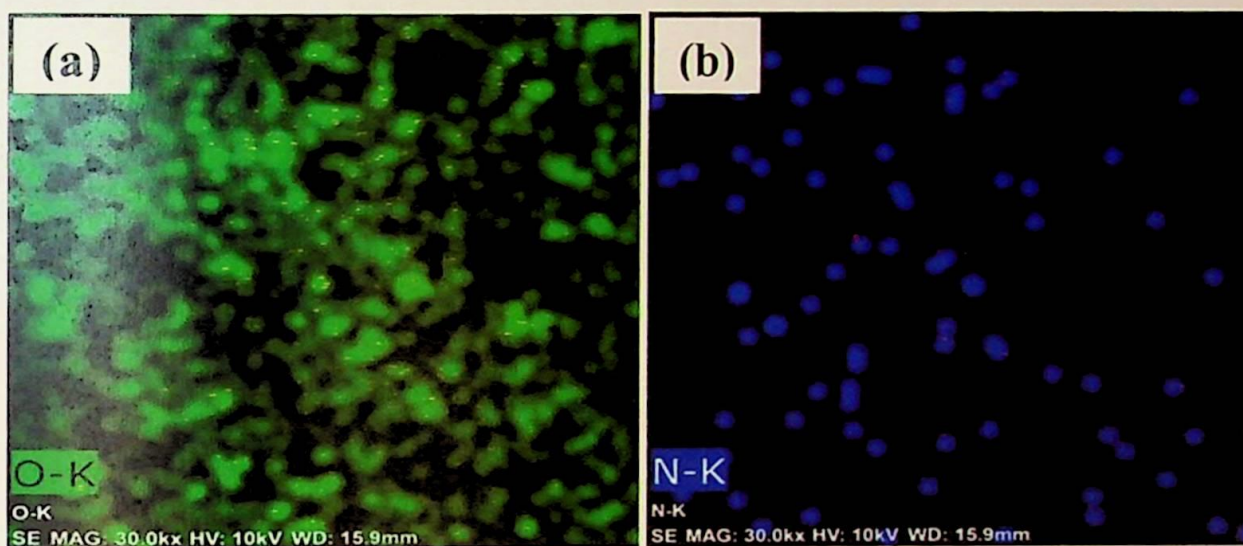


Fig. 4.5 (a) O-K lines, (b) N-K lines, (c) C-K lines for Sample C.

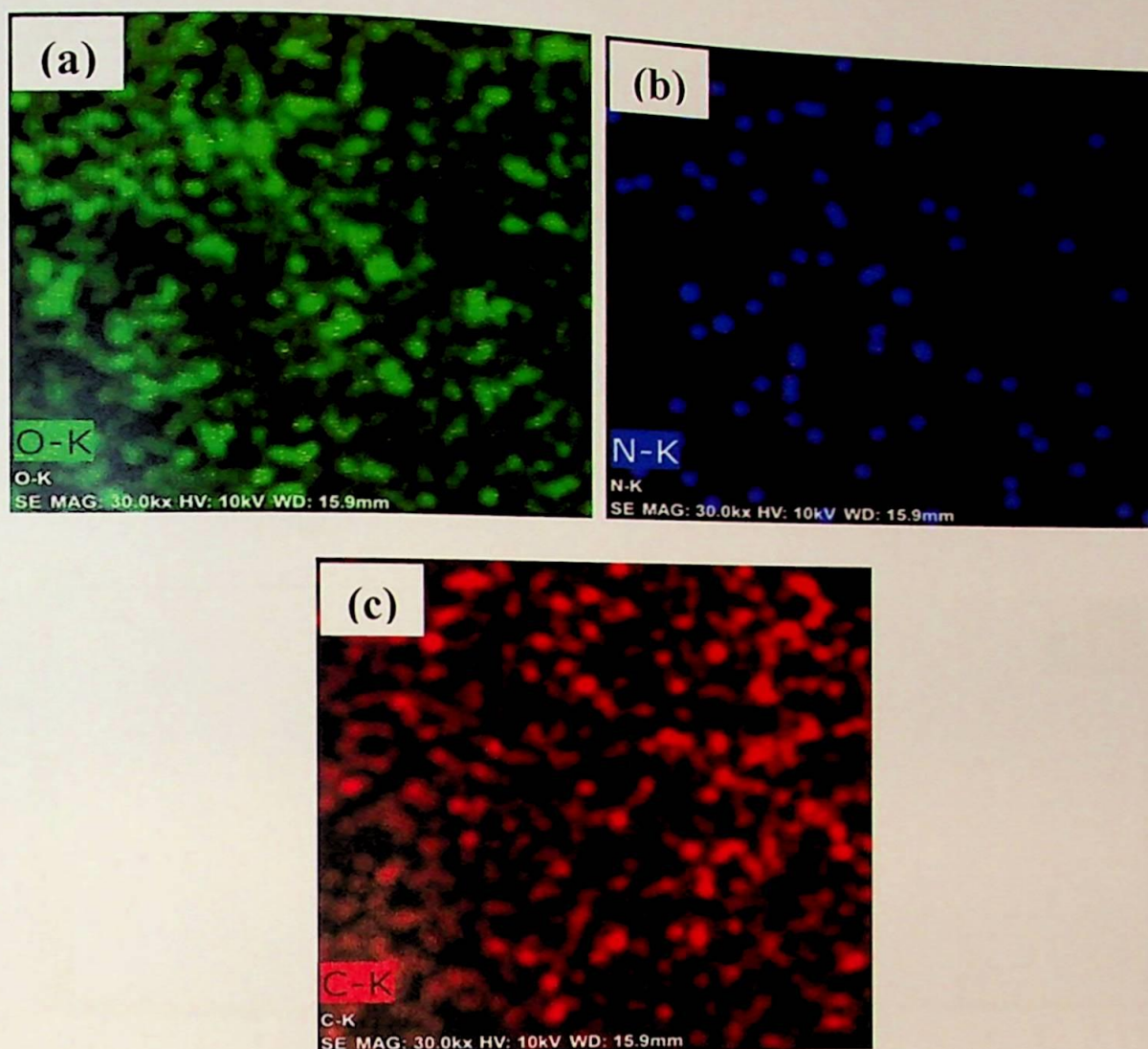


Fig. 4.6 (a) O-K lines, (b) N-K lines, (c) C-K lines for Sample D.

EDX elemental mapping detected K-shell ($K\alpha$) X-ray emissions of Oxygen (O-K), Nitrogen (N-K), and Carbon (C-K), providing spatial distribution and elemental composition of adsorbent polymers, where the green area shows uniform distribution of oxygen atoms. Even distribution of oxygen atoms suggests the presence of hydroxyl groups in Samples A, B, C, and D.

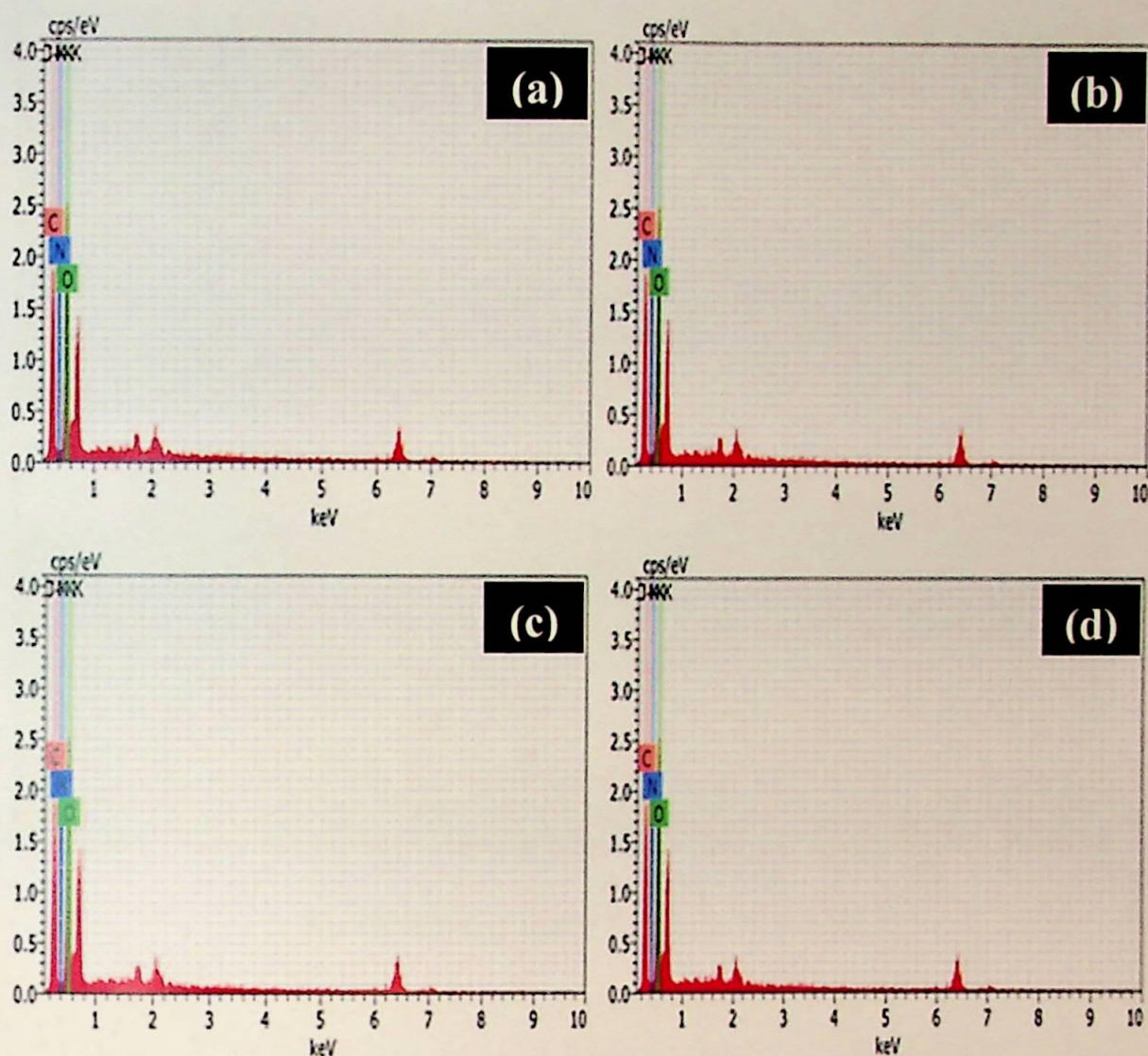


Fig. 4.7 Energy-Dispersive X-ray Spectroscopy (EDX) Analysis result for samples A, B, C and D.

Nitrogen appears as distinct, well-dispersed spots, indicating the presence of nitrogen-containing groups. Which are present as discrete domains, as grafted chains or clusters within the polymer matrix. This distribution is not uniform like carbon and oxygen, suggesting localized functionalization in samples A, B, C, and D.

The widespread red region indicates the presence of carbon atoms. Uniform carbon content suggests the organic polymer chains. This texture appears granular or porous.

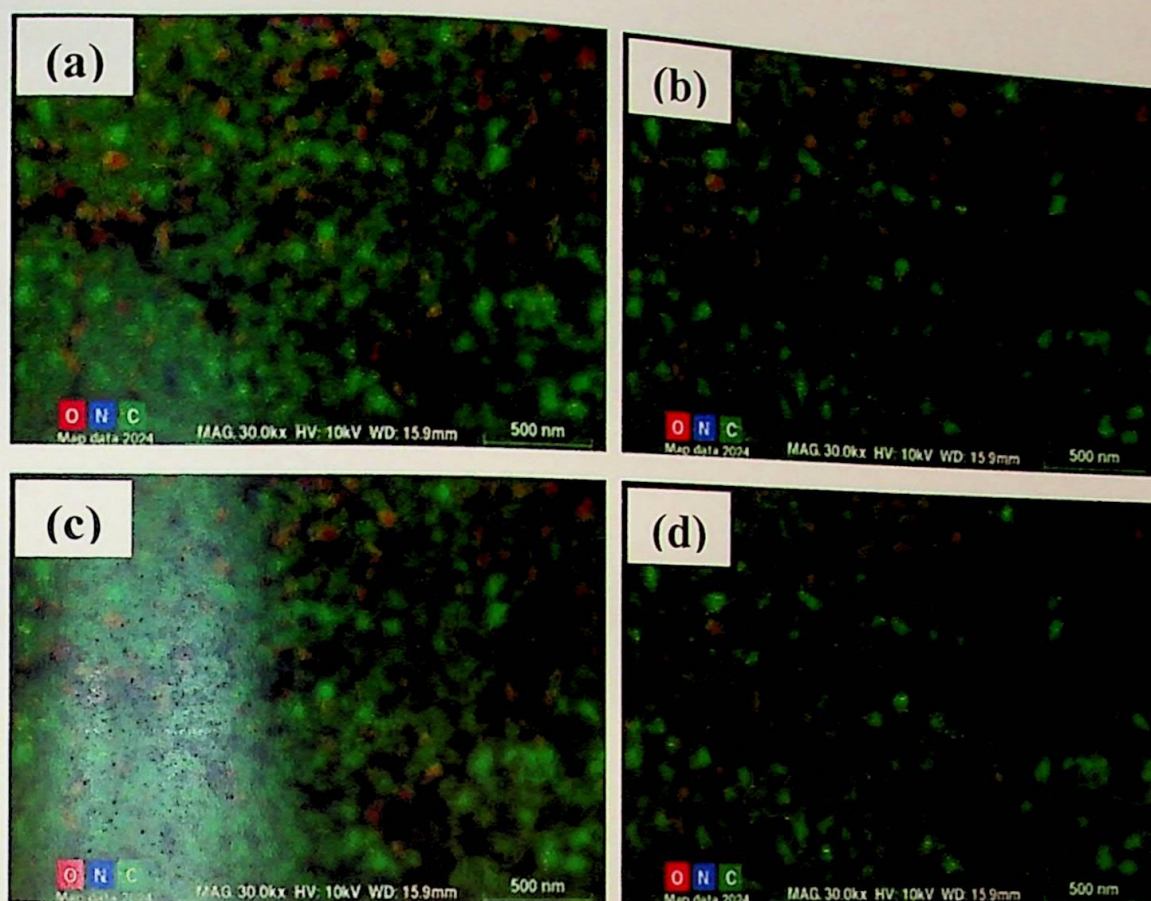


Fig. 4.8 SEM micrographs for samples A, B, C and D.

Table 4.2 Size Calculation for Samples A, B, C and D.

(Scale bar = 500nm)

Parameters

		Value	Standard Error
Counts	y0	0.45236	0.84244
	xc	53.36186	1.37079
	A	456.2628	72.18951
	w	32.44744	4.26232

Scanning Electron Microscopy (SEM) images were captured at a magnification of 30,000 $\times$ using an accelerating voltage of 10.0 kV to study the surface morphology of the adsorbent material. The polymeric adsorbent exhibited a well-defined three-dimensional mesoporous framework with an average pore diameter of nearly 53.36 nm.

4.4 Metal and Dye Absorption

Gelatin grafted copolymer was prepared and cross-linked with monomers (vinyl acetate and maleic acid) to get poly(Gelatin-g-VAc-co-MA), which was treated with

copper sulphate pentahydrate, Nickel chloride, Strontium chloride hexahydrate, and methylene blue along with a buffer solution of pH-4 [59].

4.4.1 Determination of λ_{max} of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

Fig. 4.9 shows λ_{max} of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is 720nm.

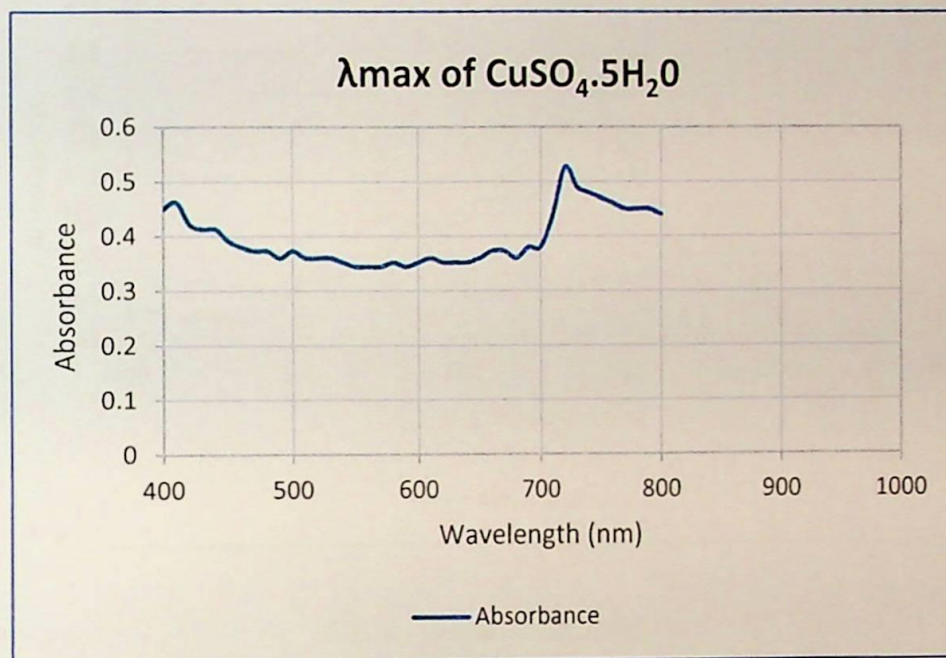


Fig. 4.9 λ_{max} of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

4.4.2 Determination of λ_{max} of NiCl_2

Fig. 4.10 shows λ_{max} of NiCl_2 is 396nm.

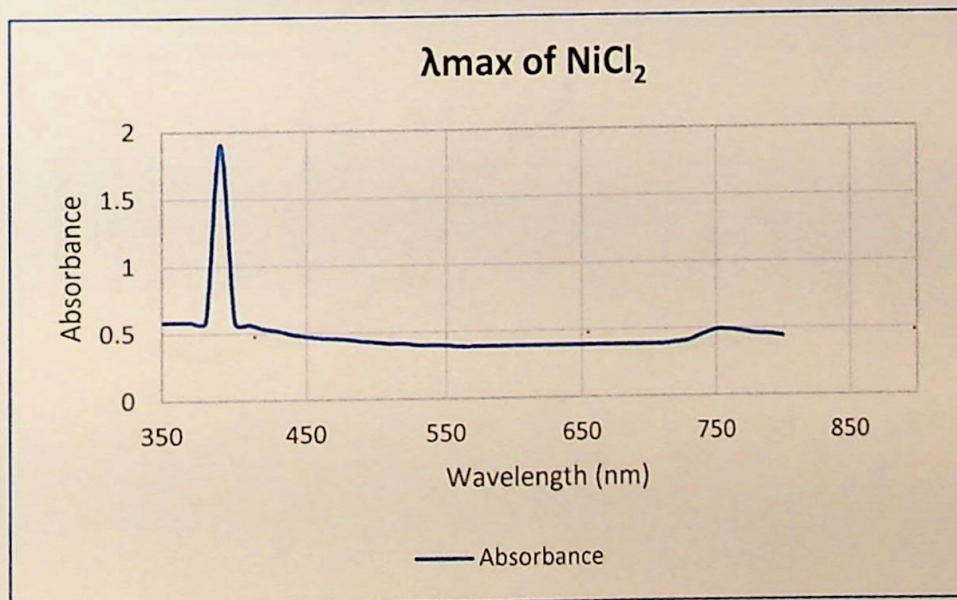


Fig. 4.10 λ_{max} of NiCl_2 .

4.4.3 Determination of λ_{max} of $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$

Fig. 4.11 shows λ_{max} of $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ is 687nm.

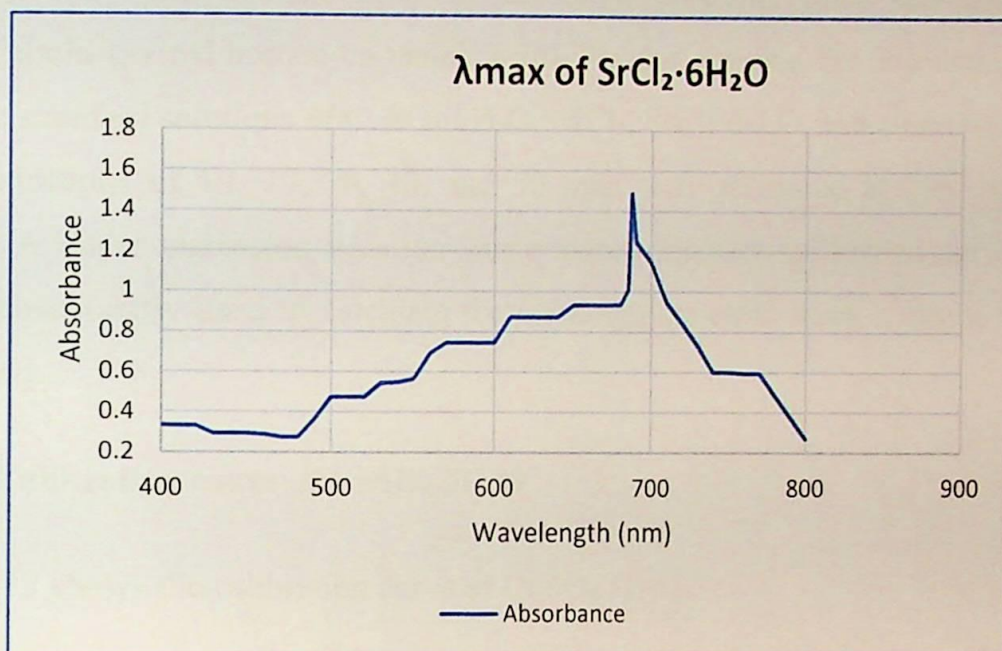


Fig. 4.11 λ_{max} of $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$.

4.4.4 Determination of λ_{max} of Methylene blue

Fig. 4.12 shows λ_{max} of Methylene blue is 665nm.

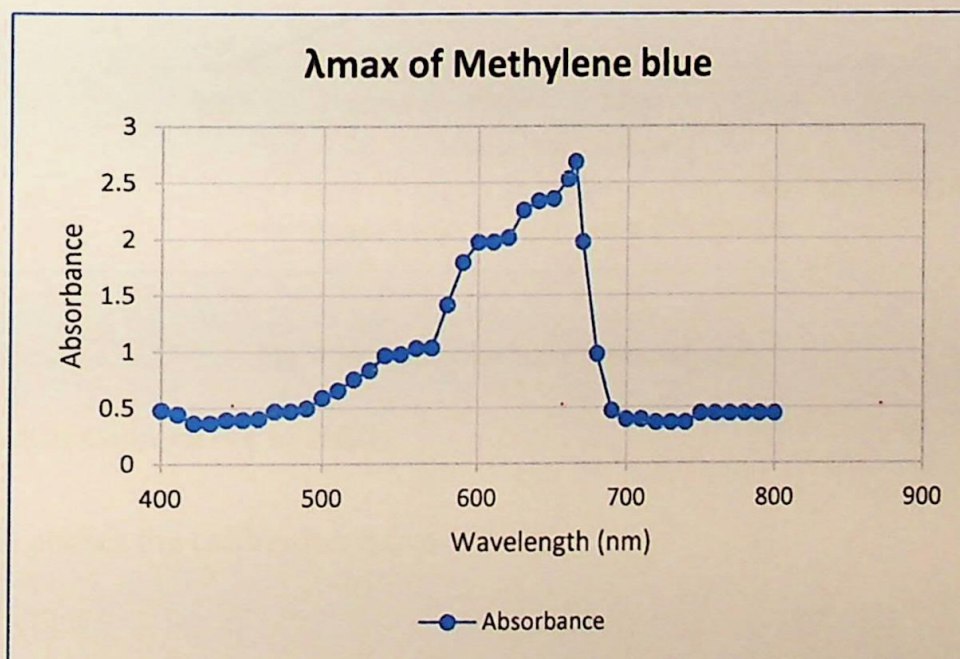


Fig. 4.12 λ_{max} of Methylene Blue.

Calibration Curves

Calibration curves were developed for Cu^{2+} , Ni^{2+} , Sr^{2+} ions, and methylene blue dye to determine the concentration of unknown solutions after adsorption onto poly(gelatin-g-vinyl acetate-co-maleic acid). For this purpose, the absorbance values of five standard solutions of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, NiCl_2 , $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$, and methylene blue at concentrations of 10, 20, 30, 40, and 50 ppm were measured at their respective λ_{max} . A linear regression equation was obtained for each calibration curve, which was subsequently used to calculate the unknown concentrations of metal ions and dye [60].

4.4.5 Calibration curve of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

Fig. 4.13 shows the calibration curve of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

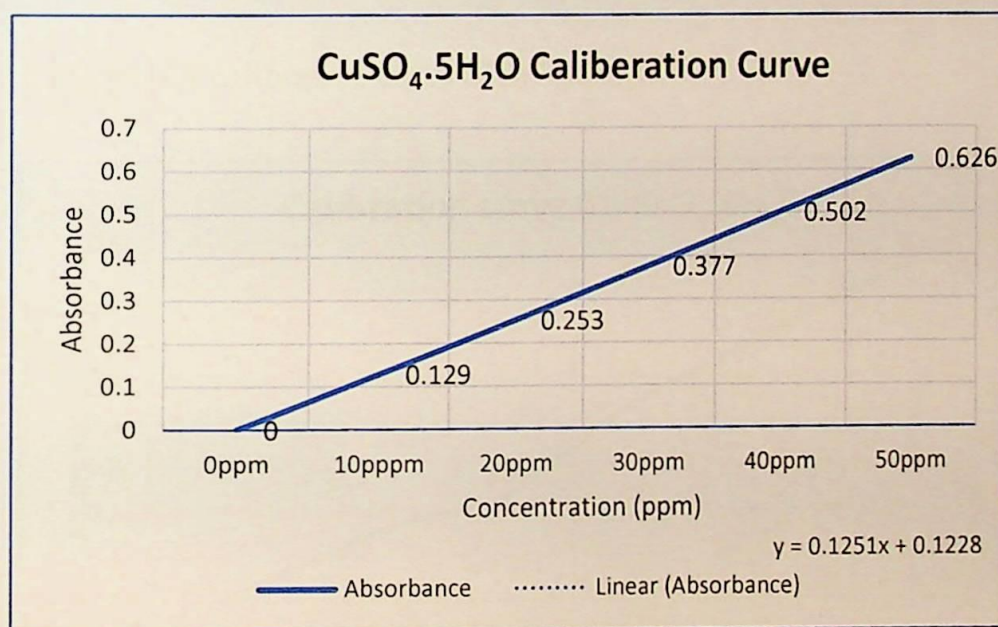


Fig. 4.13 Calibration curve for $\text{CuSO}_4 \cdot \text{H}_2\text{O}$.

4.4.6 Calibration curve of NiCl_2

Fig. 4.14 shows the calibration curve of NiCl_2 .

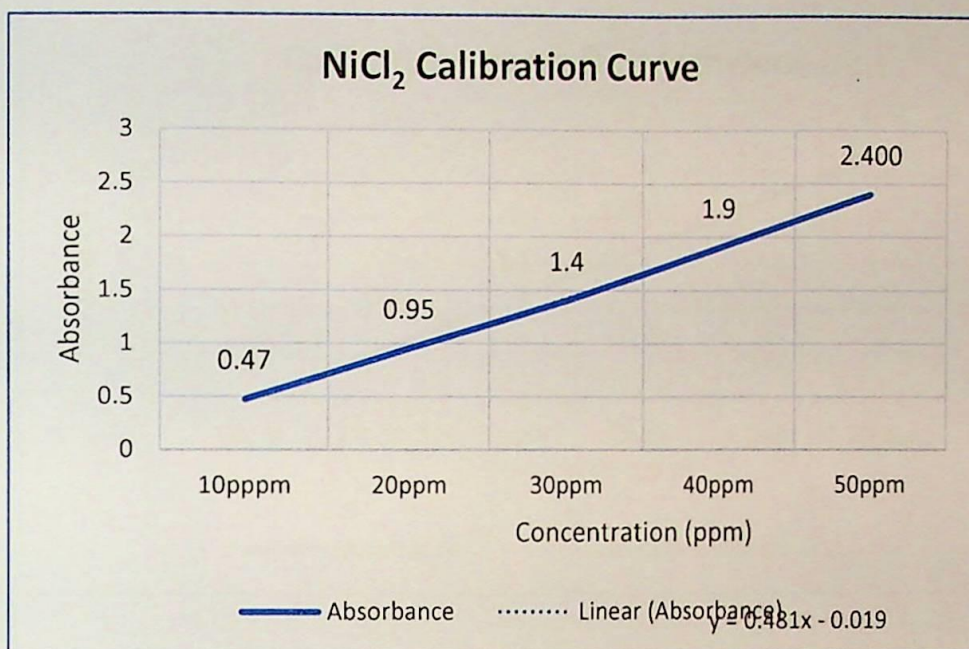


Fig. 4.14 Calibration curve for NiCl₂.

4.4.7 Calibration curve of SrCl₂.6H₂O

Fig. 4.15 shows the calibration curve of SrCl₂.6H₂O.

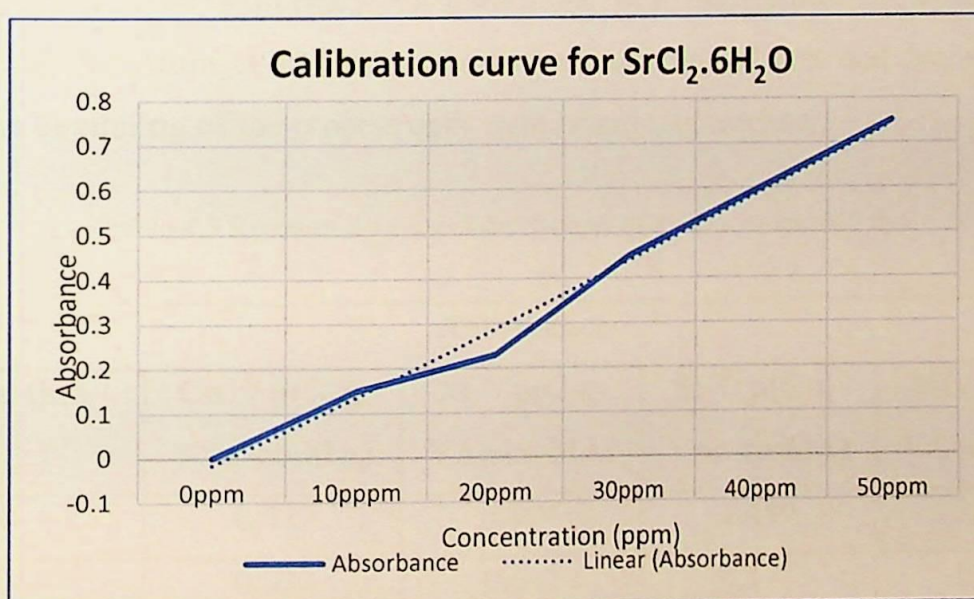


Fig. 4.15 Calibration curve for SrCl₂.6H₂O.

4.4.8 Calibration curve of Methylene blue

Fig. 4.16 shows the calibration curve of methylene blue.

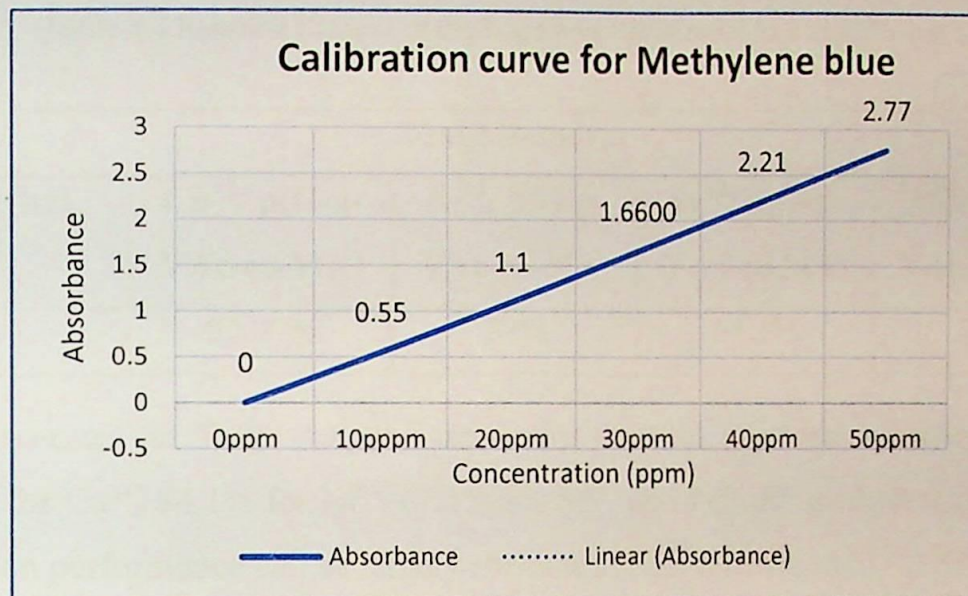


Fig. 4.16 Calibration curve for Methylene blue.

4.4.9. Reaction Kinetics

The adsorption kinetics of Cu^{2+} , Ni^{2+} , Sr^{2+} ions and methylene blue dye were evaluated by monitoring their removal from aqueous solutions using poly(Gelatin-g-VAc-co-MA). The efficiency of adsorption was determined in terms of the percentage reduction in absorbance of the respective metal ions and dye, measured from the beginning of the process until equilibrium was reached.

Table 4.3 Reaction Kinetics of the removal of metal ions and MB dye.

Time (hr)	Absorbance			
	$\text{Cu}^{+2}/ \text{p(G-g-VAc-co-MA)}$	$\text{Ni}^{+2}/ \text{p(G-g-VAc-co-MA)}$	$\text{Sr}^{+2}/ \text{p(G-g-VAc-co-MA)}$	$\text{MB}/ \text{p(G-g-VAc-co-MA)}$
0	0.527	1.904	1.489	2.684
3	0.037	0.042	0.177	0.560

Table 4.3 indicates that, independent of the type of metal ion, the adsorption kinetics for the removal of Cu^{2+} , Ni^{2+} , Sr^{2+} , and methylene blue are strongly influenced by the functional groups present in poly(Gelatin-g-VAc-co-MA). The presence of -OH and -COOH groups within the polymer matrix enhances its affinity toward both metal ions and dye molecules, thereby facilitating effective adsorption.

Table 4.4 Reaction Kinetics of the %age removal of metal ions and Mb dye.

%age Removal				
Time (hr)	$\text{Cu}^{+2}/ \text{p(G-g-VAc-co-MA)}$	$\text{Ni}^{+2}/ \text{p(G-g-VAc-co-MA)}$	$\text{Sr}^{+2}/ \text{p(G-g-VAc-co-MA)}$	$\text{MB}/ \text{p(G-g-VAc-co-MA)}$
3	92.97%	97%	88.1%	79%

As summarized in Table 4.4, the maximum removal efficiencies achieved were 75.86% for Cu^{2+} , 84.1% for Ni^{2+} , 67.1% for Sr^{2+} , and 74% for methylene blue. This adsorption performance can be largely attributed to the interaction of -COOH groups with metal ions and dye molecules. The results confirm that poly(Gelatin-g-VAc-co-MA) serves as an efficient adsorbent, offering significant potential for the treatment of wastewater contaminated with heavy metals and organic dyes.

Cu^{2+} [71], whereas chitosan-MA systems may reach 180-200 mg/g under optimized conditions [72]. Positioning the present hydrogel against such quantitative benchmarks is essential for assessing its adsorption efficiency.

Despite these advantages, some intrinsic limitations warrant attention. The acetate groups from VAc enhance mechanical integrity and suppress premature hydrogel dissolution but simultaneously increase hydrophobicity, thereby retarding water uptake and swelling kinetics. This introduces a trade-off between structural robustness and adsorption rate. Furthermore, the gelatin backbone remains biodegradable, making the material susceptible to hydrolytic cleavage and microbial degradation during prolonged aqueous exposure [73].

Another concern is competitive adsorption in multicomponent systems. In real wastewater, divalent cations such as Ca^{2+} and Mg^{2+} , as well as anions like SO_4^{2-} , may compete for active sites, thereby diminishing adsorption selectivity. Moreover, reusability and regeneration performance were not examined in this study. Previous reports indicate that gelatin- and chitosan-based hydrogels typically lose significant capacity after 3-5 adsorption-desorption cycles [74, 75]. Hence, the long-term operational stability of poly(Gelatin-g-VAc-co-MA) remains unresolved. Scale-up feasibility, grafting uniformity, and production economics also pose potential challenges for industrial translation.

Beyond aqueous metal ion and dye remediation, the physicochemical versatility of this copolymer makes it relevant for broader applications. Its biocompatibility and hydrogel-forming capacity render it suitable for biomedical uses such as wound dressings, tissue engineering scaffolds, and drug delivery matrices [76-78]. Additionally, VAc-MA based copolymers are incorporated into detergent and cleaning formulations owing to their dispersant and chelating capabilities [79]. These diverse applications highlight the multifunctional potential of the present hydrogel system.

To advance towards industrial application, several directions are recommended: Determination of maximum adsorption capacities (mg/g) for Cu^{2+} , Ni^{2+} , Sr^{2+} , and

methylene blue under batch and continuous-flow conditions. Systematic regeneration studies to evaluate adsorption performance over multiple cycles. Testing under real wastewater conditions containing mixed ionic and organic species. Optimization of crosslink density to balance swelling dynamics with mechanical stability. Pilot-scale experiments and techno-economic analysis to assess commercial viability.

Overall, poly(Gelatin-g-VAc-co-MA) demonstrates clear structural, thermal, and functional advantages compared to gelatin-PVA and gelatin-PMAA analogues. Its mesoporous morphology (~53 nm), multi-functional groups, and thermal stability up to 300 °C indicate strong potential for wastewater remediation, particularly for Cu^{2+} , Ni^{2+} , Sr^{2+} , and methylene blue. Nonetheless, challenges such as biodegradation, competitive ion effects, and regeneration efficiency must be addressed. With appropriate optimization and benchmarking, this hydrogel can evolve into a cost-effective, efficient, and versatile adsorbent for environmental cleanup, while simultaneously offering opportunities in biomedical and industrial sectors.

CONCLUSIONS

The results demonstrate that poly(Gelatin-g-VAc-co-MA) is an efficient, thermally stable, and eco-friendly adsorbent polymer, providing a cost-effective and versatile approach for the removal of heavy metal ions such as Cu^{2+} , Ni^{2+} , Sr^{2+} , as well as organic dyes from wastewater.

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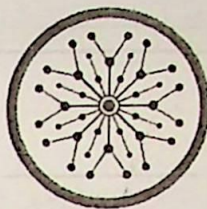
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