Synthesis and Application of Crosslinked Pectin Beads for Aluminium Sorption from Aqueous Solution

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ABSTRACT

Heavy metal contamination of water has become one of the major environmental concerns due to rapid industrialization. Among various heavy metals, aluminium has gained considerable attention because of its toxic effects on human health and the environment. The present study describes the synthesis of crosslinked pectin beads using ammonium persulfate (APS) as an initiator and N,N-methylene bisacrylamide (N,N-MBAAm) as a crosslinking agent for aluminium ion sorption from aqueous solution. The synthesized crosslinked pectin networks were evaluated for swelling behaviour, aluminium sorption efficiency and structural characterization by Fourier Transform Infrared (FTIR) spectroscopy. Swelling studies revealed that pectin-cl-N,N-MBAAm-5.0% exhibited a maximum swelling response of 585% at 6 h, whereas pectin-cl-N,N-MBAAm-1.0% showed a maximum swelling response of 412%. Aluminium sorption increased with increasing contact time and temperature. The maximum aluminium uptake of 60.32% was obtained at 55°C after 24 h using the crosslinked pectin network. FTIR analysis confirmed successful modification of pectin and indicated the interaction of aluminium ions with the functional groups present in the polymer matrix. The results demonstrate that crosslinked pectin is an efficient and economical biosorbent for aluminium removal from aqueous solution and may serve as a potential alternative to conventional adsorbents.

Keywords: Pectin, Aluminium, Biosorption, Crosslinked polymer, Swelling behaviour, FTIR, Heavy metal removal, Water treatment

INTRODUCTION

The rapid growth of industrialization and urbanization has resulted in the continuous discharge of heavy metals into aquatic environments, posing a serious threat to ecosystems and public health. Unlike many organic contaminants, heavy metals are non-biodegradable and can accumulate in living organisms through bioaccumulation and biomagnification. Industrial effluents generated from mining, electroplating, metal finishing, battery manufacturing, textile processing, paper production, and chemical industries frequently contain toxic metal ions that contaminate natural water resources. Consequently, the development of efficient, economical, and environmentally sustainable technologies for heavy-metal removal has become an important research priority[1,2,3,4]. Among the various metal pollutants, aluminium has attracted increasing attention because of its widespread industrial use and its potential adverse effects on human health and the environment. Aluminium is the third most abundant element in the Earth's crust and is extensively used in construction materials, food packaging, pharmaceuticals, cosmetics, water-treatment chemicals, and household products. Although aluminium is naturally present in soil and water, industrial activities and excessive use of aluminium-containing chemicals can significantly increase its concentration in aquatic systems. Elevated aluminium exposure has been associated with neurological disorders, dialysis encephalopathy, bone diseases, and impaired plant growth, particularly under acidic conditions where aluminium becomes more bioavailable. Therefore, effective removal of aluminium ions from contaminated water remains an important environmental challenge [5,6,7,8]. Several conventional methods have been employed for the removal of dissolved metal ions from wastewater, including chemical precipitation, ion exchange, membrane filtration, electrochemical treatment,

solvent extraction, and reverse osmosis. Although these technologies can achieve satisfactory removal efficiencies, many of them suffer from limitations such as high operational costs, complex process design, significant energy requirements, production of secondary sludge, and reduced efficiency at low metal-ion concentrations. These drawbacks have stimulated considerable interest in biosorption, an environmentally friendly alternative that utilizes naturally derived materials possessing abundant functional groups capable of binding metal ions through ion exchange, electrostatic attraction, or surface complexation [1,9]. Natural polysaccharides have emerged as attractive biosorbents because of their renewable origin, biodegradability, low toxicity, and rich surface chemistry. Among these materials, pectin has received considerable attention owing to its high content of galacturonic acid residues containing carboxyl and hydroxyl groups that readily interact with metal ions. Commercial pectin, mainly obtained from citrus peels and apple pomace, possesses excellent film-forming ability, biocompatibility, and hydrophilicity, making it useful in food, pharmaceutical, biomedical, and environmental applications. In recent years, pectin has been investigated not only as a food hydrocolloid but also as a promising adsorbent for heavy metals, dyes, pharmaceuticals, and other emerging contaminants[10,11]. Despite these advantages, native pectin exhibits relatively poor mechanical stability and dissolves or swells excessively in aqueous media, restricting its direct application in wastewater treatment. Moreover, the limited structural stability of uncrosslinked pectin often results in reduced adsorption efficiency and poor reusability. To overcome these limitations, various chemical and physical modification strategies have been explored, including ionic crosslinking, graft copolymerization, interpenetrating polymer networks, and covalent crosslinking using multifunctional agents. Such modifications improve the structural integrity of pectin, enhance pore formation, increase the accessibility of active binding sites, and improve adsorption performance[12, 13]. Among the available modification approaches, free-radical crosslinking using ammonium persulfate (APS) as an initiator and *N,N*-methylene bisacrylamide (MBAAm) as a bifunctional crosslinking agent provides an effective strategy for constructing three-dimensional polymeric networks. Crosslinking introduces additional structural stability while maintaining the abundant oxygen-containing functional groups responsible for metal-ion binding. Previous studies have demonstrated that the adsorption behaviour of pectin-based materials depends on factors such as crosslinking density, porosity, surface functional groups, solution pH, temperature, and contact time. The predominant adsorption mechanisms include electrostatic attraction, ion exchange, coordination between metal ions and carboxyl groups, and the classical "egg-box" interaction characteristic of pectin-based materials [1]. Although numerous studies have investigated pectin-based materials for the removal of heavy metals such as lead, cadmium, copper, chromium, nickel, and zinc, comparatively fewer reports have examined APS/MBAAm-crosslinked pectin systems for aluminium adsorption. Furthermore, understanding how crosslinking influences swelling behaviour and the accessibility of functional groups remains important for the rational design of efficient pectin-based biosorbents [14]. Therefore, the present study aims to synthesize crosslinked pectin using ammonium persulfate as the free-radical initiator and *N,N*-methylene bisacrylamide as the crosslinking agent and to evaluate its applicability for aluminium removal from aqueous solution. The synthesized materials were characterized by Fourier transform infrared spectroscopy (FTIR), while their swelling behaviour and aluminium sorption performance were investigated under different contact times and temperatures. The findings provide useful information regarding the influence of covalent crosslinking on the adsorption behaviour of pectin-based materials and support the development of sustainable biopolymer adsorbents for water purification.

MATERIALS AND METHODS

Materials

Pectin (food grade) was procured from Nice Chemicals Pvt. Ltd. (Kochi, India). Ammonium persulfate (APS) was purchased from CDH Pvt. Ltd. (New Delhi, India), while *N,N*-methylene bisacrylamide (MBAAm) was obtained from Loba Chemie Pvt. Ltd. (Mumbai, India). Aluminium chloride ($AlCl_3$), ethylenediaminetetraacetic acid (EDTA), zinc sulfate ($ZnSO_4$), Eriochrome Black T (EBT), acetone, and all other analytical-grade chemicals were used without further purification. Double-distilled water was used throughout the experiments.

Preparation of Crosslinked Pectin

Crosslinked pectin networks were synthesized through free-radical polymerization using ammonium persulfate (APS) as the initiator and *N,N*-methylene bisacrylamide (MBAAm) as the crosslinking agent. Initially, 1.0 g of pectin was dissolved completely in distilled water under continuous stirring until a homogeneous solution was

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obtained. Subsequently, MBAAm was added at concentrations of 1.0% and 5.0% (w/w, with respect to pectin). Thereafter, 1.0% APS was introduced into the reaction mixture to initiate free-radical crosslinking. The reaction mixture was maintained at **65 °C** for **2 h** under static conditions to allow complete crosslinking. After completion of the reaction, the insoluble polymeric product was separated by filtration and washed repeatedly with acetone to remove residual monomers and unreacted chemicals. The product was then thoroughly washed with distilled water and dried in a hot-air oven at **45 °C** until a constant weight was achieved.

The synthesized polymers were designated as:

- **Pectin-cl-MBAAm-1%**
- **Pectin-cl-MBAAm-5%**

Swelling Studies

The swelling behaviour of the synthesized crosslinked pectin samples was evaluated in distilled water at room temperature. Approximately **0.10 g** of each dried polymer sample was immersed in **50 mL** of distilled water.

At predetermined time intervals (1, 2, 4, 6, and 24 h), the swollen samples were removed, gently blotted with filter paper to eliminate excess surface water, and immediately weighed.

The percentage swelling was calculated using:

$$\text{Swelling response} = \frac{Ws - Wd}{Wd} \times 100$$

Where W_d the is the weight of dry sample and W_s is the weight of wet sample.

Aluminium Adsorption Studies

Aluminium adsorption experiments were performed using aqueous aluminium chloride solution. A stock solution (500 ppm) of $AlCl_3$ was prepared in double-distilled water. For each adsorption experiment, **0.25 g** of the synthesized crosslinked pectin was added to **50 mL** of aluminium solution and maintained under static conditions. To investigate the effect of contact time, aliquots (5 mL) were withdrawn after **1, 2, 4, 24, 48, and 72 h**. The residual aluminium concentration was determined by EDTA back titration using zinc sulfate as the titrant and Eriochrome Black T as the indicator.

The percentage removal of aluminium ions was calculated using:

$$\text{Percentage uptake } (P_u) = \frac{Cs - Ct}{Cs} \times 100$$

Where, C_s is the standard concentration of Al^{3+} ions and C_t is the concentration at particular time.

Effect of Temperature

The influence of temperature on aluminium adsorption was investigated at **22 °C** and **55 °C**, while maintaining all other experimental conditions constant. Aluminium removal was determined at different contact times using the procedure described above.

Fourier Transform Infrared (FTIR) Analysis

Fourier Transform Infrared (FTIR) spectroscopy was employed to characterize native pectin, crosslinked pectin, and aluminium-loaded crosslinked pectin. The spectra were recorded using an Agilent FTIR spectrophotometer over the range of **4000–400 cm^{-1}**. The obtained spectra were analyzed to identify the characteristic functional groups involved in crosslinking and aluminium adsorption.

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Scanning Electron Microscopy (SEM)

The surface morphology of the synthesized crosslinked pectin samples was examined using scanning electron microscopy (SEM). The micrographs were analyzed to evaluate surface roughness, pore formation, and morphological changes induced by crosslinking. Particular attention was given to the development of porous structures that could facilitate aluminium adsorption.

RESULTS AND DISCUSSION

Synthesis of Crosslinked Pectin

The successful preparation of crosslinked pectin was achieved through free-radical polymerization using ammonium persulfate (APS) as the initiator and *N,N*-methylene bisacrylamide (MBAAm) as the bifunctional crosslinking agent. During the polymerization process, thermal decomposition of APS generated sulfate radical anions capable of abstracting hydrogen atoms from the hydroxyl groups present along the pectin backbone. The resulting macroradicals subsequently reacted with MBAAm molecules, leading to the formation of covalent bridges between adjacent polymer chains and producing a stable three-dimensional polymeric network. This crosslinking process enhanced the structural integrity of the pectin matrix and reduced its tendency to dissolve in aqueous media. The formation of an interconnected polymer network is expected to improve the mechanical stability of pectin while simultaneously increasing the accessibility of functional groups involved in metal-ion binding. The incorporation of MBAAm introduces additional free volume within the polymer structure, facilitating the penetration of water molecules and improving the diffusion of aluminium ions into the interior of the adsorbent. Similar free-radical crosslinking strategies have been reported for polysaccharide-based hydrogels, where increased network rigidity and improved structural stability contributed to enhanced adsorption performance and swelling characteristics. Figure 3.1 illustrates the proposed free-radical crosslinking mechanism between pectin and MBAAm initiated by APS. Although the proposed reaction mechanism has not been confirmed experimentally by spectroscopic monitoring of reaction intermediates, the successful formation of an insoluble crosslinked material together with subsequent FTIR analysis supports the occurrence of chemical modification.

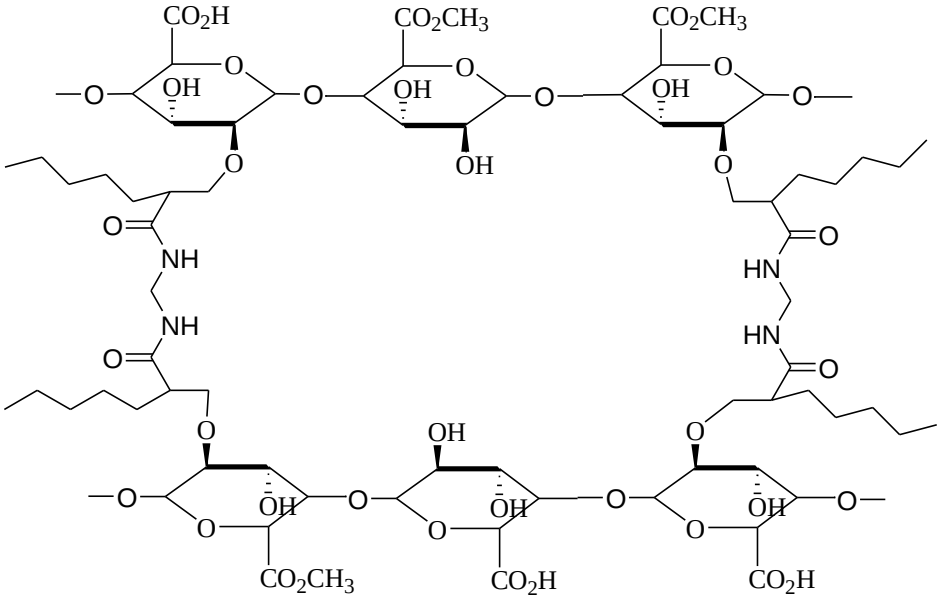

Fig. 3.1: Proposed structure for pectin-*cl*-N,N-MBAAm.

The degree of crosslinking plays a crucial role in determining the physicochemical properties of hydrogel-based adsorbents. A suitable crosslinking density improves dimensional stability while maintaining sufficient flexibility for water absorption and diffusion of metal ions. Excessive crosslinking may restrict polymer chain mobility and reduce the availability of adsorption sites, whereas insufficient crosslinking often results in poor mechanical strength and dissolution of the polymer matrix during adsorption experiments. Therefore, optimization of crosslinker concentration is essential for balancing swelling behaviour and adsorption efficiency.

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In the present study, two crosslinker concentrations (1% and 5% MBAAm) were investigated to evaluate their influence on swelling behaviour and aluminium sorption. The comparison of these two compositions provides insight into the relationship between network architecture and adsorption performance. The higher MBAAm concentration was expected to generate a more interconnected polymeric structure with improved dimensional stability and enhanced accessibility of functional groups responsible for aluminium binding. Overall, the synthesis procedure successfully produced chemically modified pectin capable of retaining its structural integrity during swelling and adsorption studies. The prepared crosslinked materials were subsequently characterized by FTIR spectroscopy, and their swelling behaviour and aluminium sorption performance were systematically evaluated under different experimental conditions.

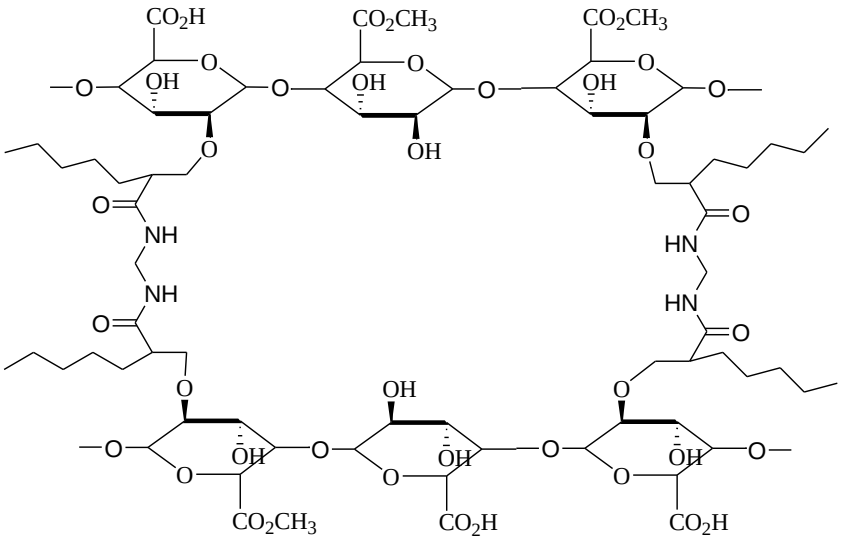

Fig. 3.1: Proposed structure for pectin-*cl*-N,N-MBAAm.

Swelling Behaviour of Crosslinked Pectin

The swelling behaviour of polymeric adsorbents is one of the most important parameters governing their adsorption performance because it determines the accessibility of water molecules and dissolved metal ions to the active adsorption sites within the polymer matrix. Hydrophilic polymers such as pectin absorb water through the interaction of hydroxyl and carboxyl functional groups with water molecules, resulting in expansion of the polymer network. The extent of swelling therefore provides valuable information regarding the internal structure, crosslinking density, and diffusion characteristics of the synthesized material. The swelling behaviour of **Pectin-cl-MBAAm-1%** and **Pectin-cl-MBAAm-5%** was investigated in distilled water at different immersion times, and the obtained results are summarized in Table 3.1. Both crosslinked polymers exhibited rapid water uptake during the initial hours of immersion, followed by a gradual approach towards equilibrium swelling after approximately **6 h**. This behaviour is characteristic of hydrophilic polymeric networks, where water initially diffuses rapidly into the interconnected pores owing to the large osmotic pressure difference between the dry polymer matrix and the surrounding aqueous medium. As hydration proceeds, polymer chain relaxation occurs until equilibrium is established between the osmotic driving force and the elastic restoring force of the crosslinked network. Among the two synthesized materials, **Pectin-cl-MBAAm-5%** exhibited consistently higher swelling than **Pectin-cl-MBAAm-1%** throughout the experimental period. The maximum swelling response of the 5% crosslinked polymer reached **585% after 6 h**, whereas the corresponding value for the 1% crosslinked polymer was **412%**. The greater swelling observed for the polymer containing a higher concentration of MBAAm suggests that the crosslinking process produced a stable three-dimensional network capable of retaining larger quantities of water without losing its structural integrity. The interconnected polymer chains likely generated additional free volume and water-accessible pores, thereby facilitating diffusion of water molecules throughout the hydrogel matrix.

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Table 3.1: Effect of Temperature on Swelling Response.

Time (in hours)	Swelling response (%) Pectin-*cl*-N,N-MBAAm-(1.0%)	Pectin-*cl*-N,N-MBAAm-(5.0%)
1	**80**	**81**
2	**168**	**164**
4	**404**	**578**
6	**412**	**585**
24	**136**	**148**

The observed increase in swelling with increasing crosslinker concentration may appear counterintuitive because highly crosslinked polymers often exhibit reduced swelling owing to restricted chain mobility. However, the behaviour depends not only on crosslink density but also on the architecture of the polymer network. In the present study, the incorporation of MBAAm may have promoted the formation of a more homogeneous and interconnected porous structure that enhanced water diffusion while maintaining sufficient mechanical stability. Similar observations have been reported for chemically modified polysaccharide hydrogels, where an optimum degree of crosslinking improved water absorption by preventing collapse of the polymer network during hydration rather than simply restricting chain movement. After **24 h**, both polymers exhibited a noticeable decrease in swelling compared with the maximum values recorded at **6 h**. This reduction may be attributed to structural relaxation of the swollen polymer network following equilibrium hydration. Prolonged immersion can result in partial rearrangement of polymer chains, slight contraction of swollen pores, or redistribution of absorbed water within the matrix, leading to a decrease in the measured swelling ratio. Similar equilibrium behaviour has been reported for crosslinked pectin hydrogels and other natural polymer-based networks, where swelling initially increases rapidly before stabilizing or decreasing slightly because of network relaxation. The enhanced swelling behaviour of the crosslinked polymers is expected to play an important role in aluminium adsorption. Increased water uptake facilitates transport of dissolved aluminium ions into the interior of the polymer network, thereby improving contact between the metal ions and the carboxyl groups of galacturonic acid residues that serve as the principal adsorption sites. Consequently, polymers exhibiting greater swelling generally provide improved accessibility of functional groups, which can contribute to higher adsorption efficiency. The swelling behaviour observed in the present investigation demonstrates that crosslinking successfully modified the physicochemical properties of native pectin while preserving its hydrophilic nature. The improved water absorption capacity, particularly for **Pectin-cl-MBAAm-5%**, indicates that the synthesized material possesses a favourable porous network suitable for subsequent aluminium adsorption studies. These findings also support the hypothesis that appropriate chemical crosslinking can significantly improve the structural stability and functional performance of pectin-based biosorbents in aqueous environments.

Aluminium Sorption Studies

Effect of Contact Time on Aluminium Adsorption

Table 3.2: Percent uptake of Al^{3+}at 22^0C Temperature.

Time(in hours)	Percent uptake Pectin	Pectin-*cl*-N,N-MBAAm
2	6	6
4	6.6	7
24	6.6	10

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48	14	16
72	16	20

Effect of Temperature

The influence of temperature on aluminium adsorption was evaluated at **55°C**, and the results are summarized in **Table 3**.

The adsorption efficiency increased with increasing temperature, indicating that elevated temperature enhances the interaction between aluminium ions and the active functional groups present in the polymer network.

The maximum aluminium uptake of **60.32%** was obtained for pectin-cl-N,N-MBAAm after **24 h** at **55°C**, whereas native pectin exhibited comparatively lower adsorption under the same experimental conditions.

The enhanced adsorption at higher temperature may be attributed to increased mobility of aluminium ions, improved diffusion into the polymer network, and greater accessibility of adsorption sites. Elevated temperature may also promote expansion of the polymer matrix, thereby exposing additional carboxyl groups responsible for aluminium binding.

The overall adsorption behaviour demonstrates that crosslinked pectin possesses considerably better adsorption efficiency than unmodified pectin.

The adsorption of aluminium ions onto pectin and crosslinked pectin was investigated as a function of contact time at **22 °C**, and the results are presented in Table 3.2. Contact time is an important parameter in adsorption studies because it determines the duration available for interaction between the adsorbent surface and the adsorbate molecules. The adsorption profile obtained in the present study indicates that both native pectin and crosslinked pectin exhibited a gradual increase in aluminium uptake with increasing contact time, although the adsorption efficiency of the chemically modified polymer was consistently higher than that of unmodified pectin throughout the experimental period.

Table 3.2: Percent uptake of Al^{3+}at 22^0C Temperature.

Time (in hours)	Percent uptake Pectin	Pectin-*cl*-N,N-MBAAm
2	6	6
4	6.6	7
24	6.6	10
48	14	16
72	16	20

Initially, both adsorbents exhibited relatively low adsorption efficiencies, with aluminium removal ranging from **6–7%** during the first few hours of contact. The low adsorption during the initial stage may be attributed to the limited diffusion of aluminium ions into the internal polymer network and the time required for hydration of the adsorbent. As the contact time increased, aluminium uptake gradually improved, reaching **20%** for crosslinked pectin after **72 h**, whereas native pectin achieved a comparatively lower adsorption efficiency of **16%** under identical experimental conditions. These observations demonstrate that chemical modification of pectin through MBAAm crosslinking improved its ability to retain aluminium ions from aqueous solution. The superior adsorption performance of the crosslinked polymer can be attributed to the structural modifications introduced during the free-radical crosslinking process. Formation of a three-dimensional polymeric network is expected to improve the accessibility of the abundant carboxyl and hydroxyl groups present on the galacturonic acid units

of pectin. These functional groups serve as the primary adsorption sites for aluminium ions through electrostatic interactions and coordination bonding. In addition, the porous morphology generated by chemical crosslinking facilitates diffusion of the metal ions into the interior of the adsorbent, thereby increasing the probability of interaction with available binding sites. The gradual increase in adsorption with time suggests that the aluminium sorption process proceeded through progressive occupation of the available active sites. During the initial stages of adsorption, numerous vacant binding sites are available on the polymer surface, allowing relatively rapid attachment of aluminium ions. As adsorption progresses, the number of unoccupied sites decreases, leading to increased competition among metal ions and a corresponding reduction in the adsorption rate. Ultimately, the adsorption process approaches equilibrium when most accessible functional groups become occupied. Although the present study did not investigate adsorption kinetics using pseudo-first-order or pseudo-second-order models, the observed time-dependent behaviour is consistent with adsorption processes commonly reported for natural polysaccharide-based adsorbents. Future studies involving kinetic modelling would provide a more detailed understanding of the adsorption mechanism and the rate-controlling steps governing aluminium uptake. Overall, the results indicate that crosslinking successfully enhanced the adsorption performance of pectin under ambient conditions. Although the absolute removal efficiency remained moderate, the improvement observed after chemical modification demonstrates that structural stabilization of pectin contributes positively to its adsorption behaviour and highlights the potential of chemically modified biopolymers as environmentally benign adsorbents for aqueous metal-ion removal.

Effect of Temperature on Aluminium Adsorption

Temperature plays a significant role in adsorption processes because it influences both the mobility of adsorbate molecules and the physicochemical properties of the adsorbent. To evaluate the influence of temperature, aluminium adsorption experiments were performed at **55 °C**, and the results are summarized in Table 3.3. Comparison of the adsorption data obtained at **22 °C** and **55 °C** revealed a substantial improvement in aluminium removal at elevated temperature for both native and crosslinked pectin. The adsorption efficiency of the crosslinked polymer increased progressively with contact time, reaching a maximum aluminium uptake of **60.32%** after **24 h**, whereas native pectin achieved **53.00%** under the same conditions. These values are considerably higher than those observed at room temperature, indicating that elevated temperature promotes aluminium adsorption by the synthesized polymer. The enhanced performance of the crosslinked material further demonstrates the beneficial effect of chemical modification on adsorption behaviour.

Table 3.3: Percent uptake of Al^{3+}at 55^0 Temperature.

Time (in hours)	Percent uptake (%) At 55^0C Pectin (%)	Pectin-*cl*-N,N-MBAAm
1	47.00	52.86
2	52.40	51.72
3	51.90	53.00
8	52.80	55.45
24	53.00	60.32

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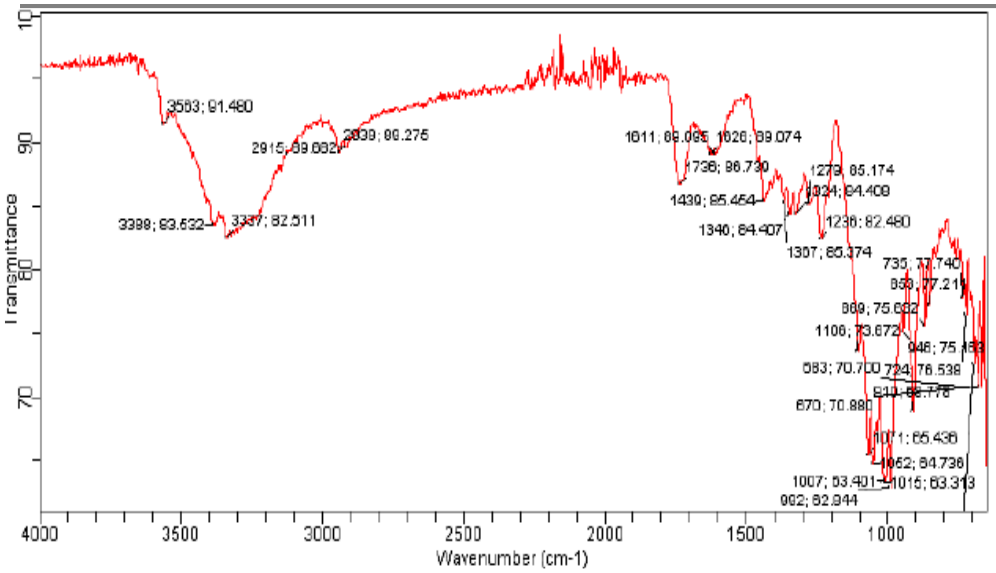
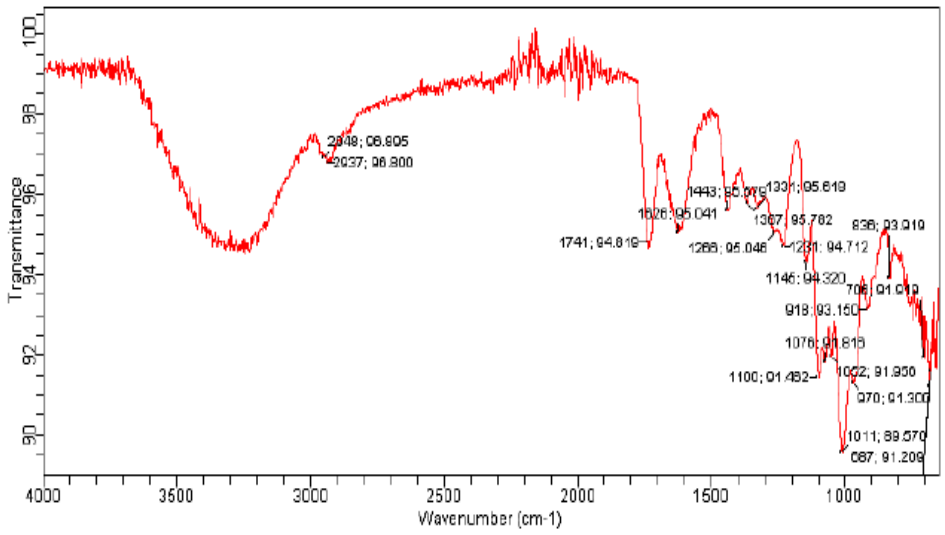


Fig. 3.2: FTIR spectrum of pectin.



The increase in adsorption with temperature may be attributed to several factors. Higher temperatures generally increase the kinetic energy of dissolved metal ions, resulting in faster diffusion from the bulk solution to the adsorbent surface. Enhanced molecular mobility facilitates penetration of aluminium ions into the interconnected polymer network and promotes interaction with the available carboxyl groups responsible for metal binding. In addition, thermal expansion of the hydrogel matrix may slightly increase the accessibility of internal adsorption sites, thereby improving the overall adsorption efficiency. The consistently higher adsorption observed for crosslinked pectin compared with native pectin indicates that the chemically modified polymer possessed a more favourable network structure for aluminium binding. Crosslinking improves dimensional stability while maintaining hydrophilic functional groups capable of coordinating with trivalent aluminium ions. Consequently, the modified adsorbent exhibits greater adsorption efficiency under both ambient and elevated temperature conditions. It should be noted that the present investigation evaluated the influence of temperature at only two experimental conditions (22 °C and 55 °C). Therefore, thermodynamic parameters such as Gibbs free energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) could not be reliably calculated. Nevertheless, the observed increase in adsorption efficiency with increasing temperature suggests that aluminium sorption by crosslinked pectin is favoured under elevated temperature conditions within the investigated range.

The combined results of the contact-time and temperature studies clearly demonstrate that chemical crosslinking significantly improves the adsorption behaviour of pectin towards aluminium ions. The enhanced adsorption performance can be attributed to the formation of a stable three-dimensional polymer network possessing abundant oxygen-containing functional groups and improved accessibility of adsorption sites. These findings

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support the potential application of crosslinked pectin as a low-cost and environmentally sustainable biosorbent for aluminium removal from aqueous media, while also indicating the need for future investigations involving adsorption kinetics, equilibrium isotherms, pH optimization, and regeneration studies to fully evaluate its practical applicability.

Fourier Transform Infrared (FTIR) Characterization

Fourier Transform Infrared (FTIR) spectroscopy was employed to investigate the structural modification of pectin following chemical crosslinking with *N,N*-methylene bisacrylamide (MBAAm) and to evaluate the interaction between the synthesized polymer and aluminium ions. FTIR analysis is a valuable technique for identifying changes in the functional groups of polysaccharides and provides indirect evidence for successful crosslinking and metal-ion coordination. The spectra of native pectin, crosslinked pectin, and aluminium-loaded crosslinked pectin are presented in **Figures 3.2–3.5**. The FTIR spectrum of native pectin exhibited characteristic absorption bands at approximately **3388 cm⁻¹**, **2939 cm⁻¹**, **1736 cm⁻¹**, and **1052 cm⁻¹**. The broad absorption band centred around **3388 cm⁻¹** corresponds to the stretching vibration of hydroxyl (–OH) groups involved in extensive intra- and intermolecular hydrogen bonding within the polysaccharide structure. The peak observed near **2939 cm⁻¹** is attributed to aliphatic C–H stretching vibrations of the pectin backbone. A strong absorption band at **1736 cm⁻¹** represents the stretching vibration of ester carbonyl (C=O) groups associated with methyl-esterified galacturonic acid residues, whereas the band around **1052 cm⁻¹** corresponds to C–O–C stretching vibrations of the glycosidic linkages characteristic of the pectin backbone (Figure 3.2). These absorption bands are consistent with the typical infrared spectral features reported for pectin and confirm the structural integrity of the native polysaccharide. Following chemical modification with MBAAm, noticeable changes were observed in the FTIR spectrum of the crosslinked polymer. A characteristic absorption band appeared at approximately **1741 cm⁻¹**, indicating modification of the polymeric structure after the crosslinking reaction (Figure 3.3). The slight shift in the carbonyl absorption band compared with native pectin suggests changes in the chemical environment of the carbonyl groups resulting from the incorporation of the crosslinking agent into the polymer network. These spectral changes provide indirect evidence that free-radical polymerization successfully altered the molecular structure of pectin and generated a chemically stabilized three-dimensional network. After adsorption of aluminium ions, additional changes were detected in the FTIR spectra of the aluminium-loaded samples. In particular, the appearance of an absorption band in the **600–700 cm⁻¹** region, with a prominent peak near **685 cm⁻¹**, indicates the interaction of aluminium ions with oxygen-containing functional groups present in the polymer matrix. This spectral feature may be attributed to Al–O stretching vibrations arising from coordination between aluminium ions and the carboxylate groups of galacturonic acid residues (Fig. 3.4.a ,3.4.b and 3.5). The observed spectral changes therefore support the participation of carboxyl functional groups as the principal adsorption sites responsible for aluminium binding.

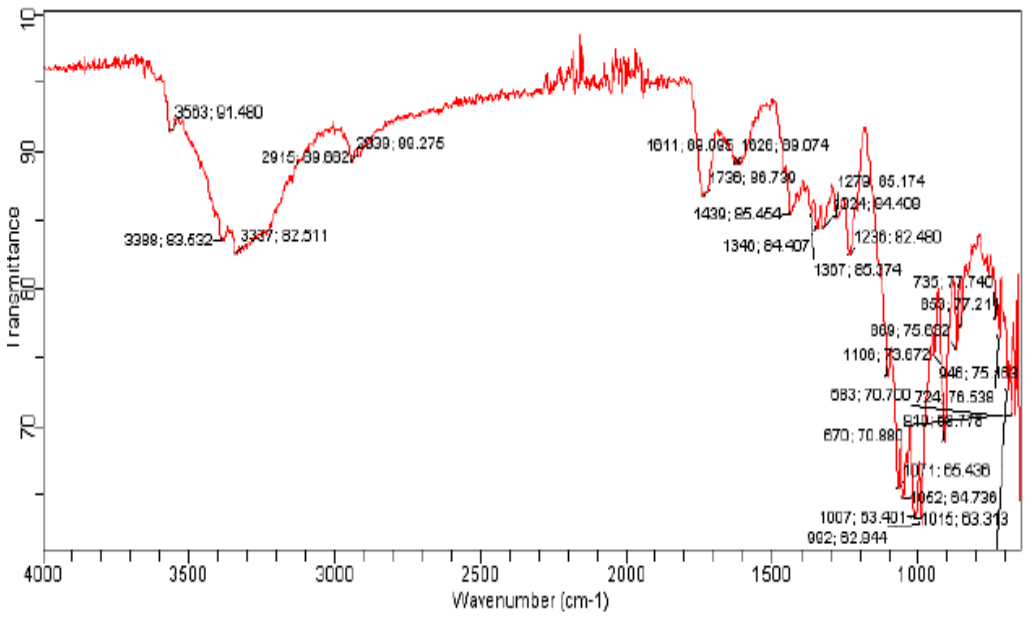

Fig. 3.2: FTIR spectrum of pectin.

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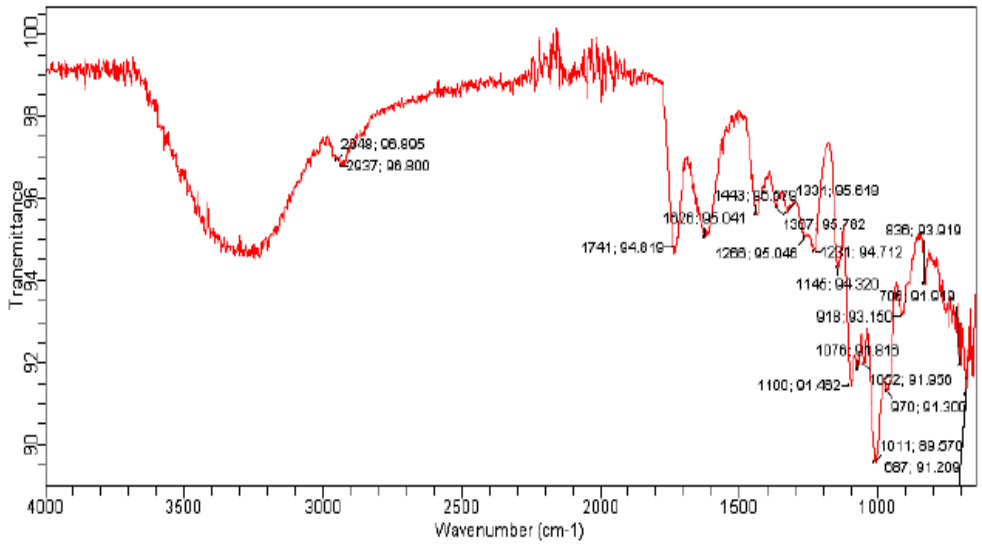
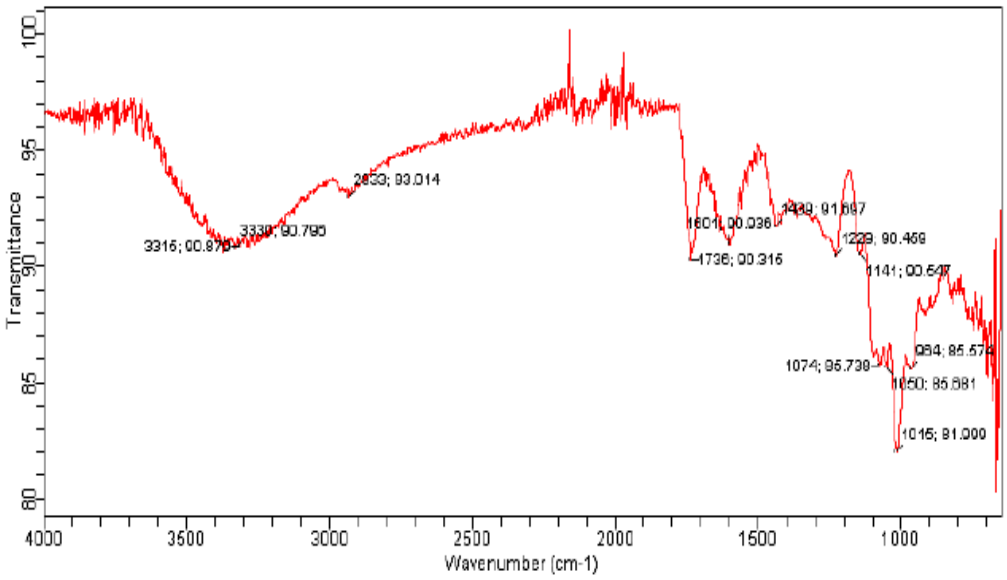


Fig. 3.3: FTIR spectrum of Pectin-*cl*-N,N-MBAAm.



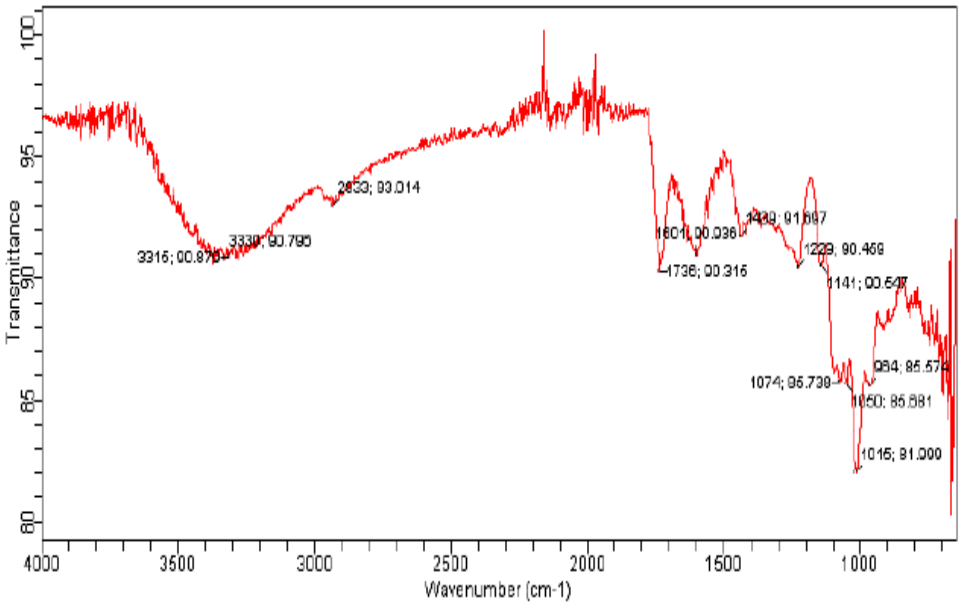


Fig. 3.4.a: FTIR spectrum of pectin at 22^0C

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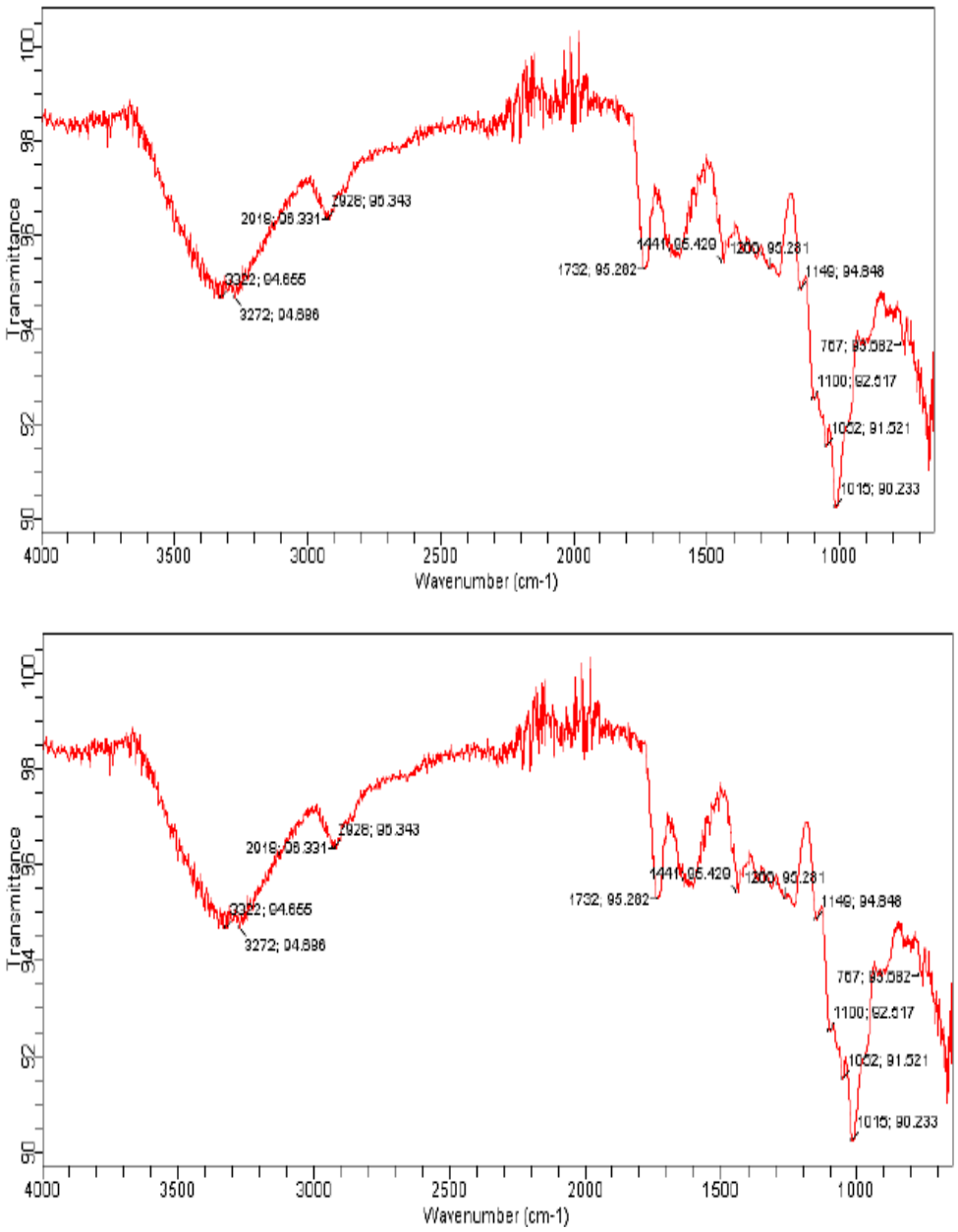
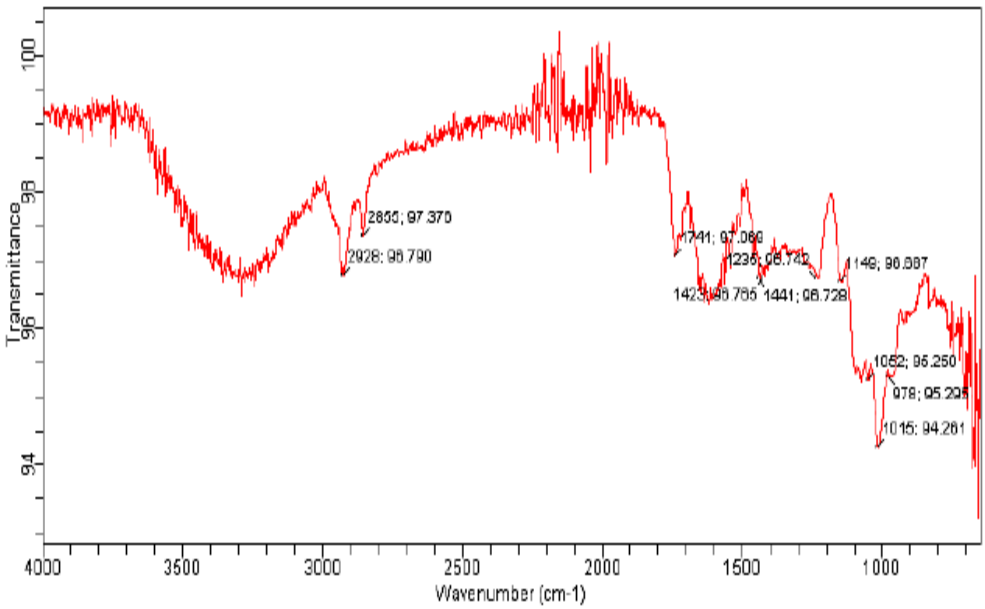


Fig.3.4.b: FTIR spectrum of Pectin-*cl*-N,N-MBAAm at 22^0C



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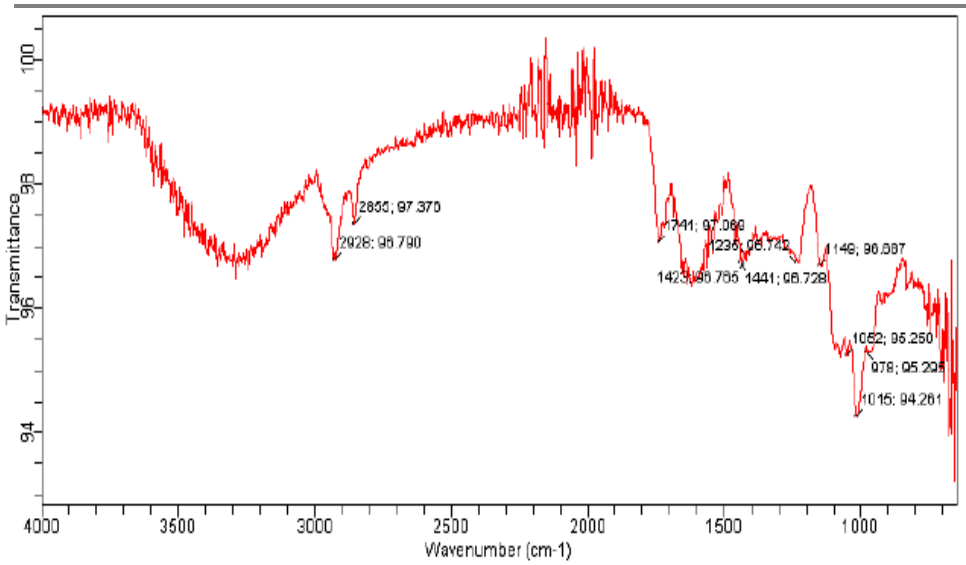
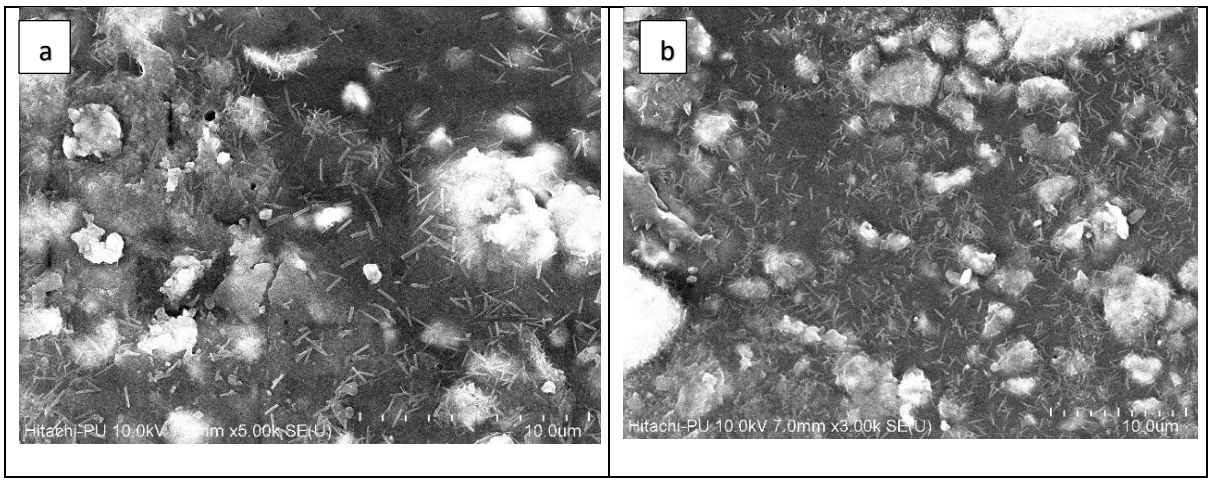


Fig.3.5: FTIR spectrum of Pectin-*cl*-N,N-MBAAm at 55^0C

The FTIR results obtained in the present study adequately demonstrate that chemical modification of pectin was successfully achieved and that the synthesized polymer possesses functional groups capable of interacting effectively with aluminium ions. Overall, the FTIR analysis confirms that the crosslinking process preserved the characteristic polysaccharide structure of pectin while introducing structural modifications that enhanced its adsorption capability. The observed spectral changes before and after aluminium adsorption provide convincing evidence that the abundant oxygen-containing functional groups of the crosslinked polymer play a dominant role in the adsorption process and contribute significantly to the improved performance of the modified biosorbent.

Surface Morphology and Proposed Aluminium Adsorption Mechanism

Scanning Electron Microscopy (SEM) was employed to investigate the surface morphology of the synthesized crosslinked pectin before aluminium adsorption. Representative SEM micrographs of **Pectin-cl-MBAAm-1% (Figure 3.6 a,b)** and **Pectin-cl-MBAAm-5% (Figure 3.6 c,d)** revealed irregular and heterogeneous surface morphologies, indicating that chemical crosslinking altered the native structure of pectin. The **Pectin-cl-MBAAm-1%** sample (**Figure 3.6 a,b**) exhibited an uneven surface with irregular domains, shallow pores, and surface roughness. In comparison, the **Pectin-cl-MBAAm-5%** sample (**Figure 3.6c,d**) displayed a relatively rougher and denser surface with more pronounced surface irregularities, suggesting the formation of a more developed crosslinked polymer network at the higher MBAAm concentration. The observed rough and heterogeneous surface morphology is expected to facilitate the interaction of aluminium ions with the adsorbent by providing accessible surface-active sites. These morphological observations are consistent with the improved swelling behaviour and adsorption performance of the crosslinked pectin. However, the interpretation remains qualitative because SEM provides only surface morphological information.



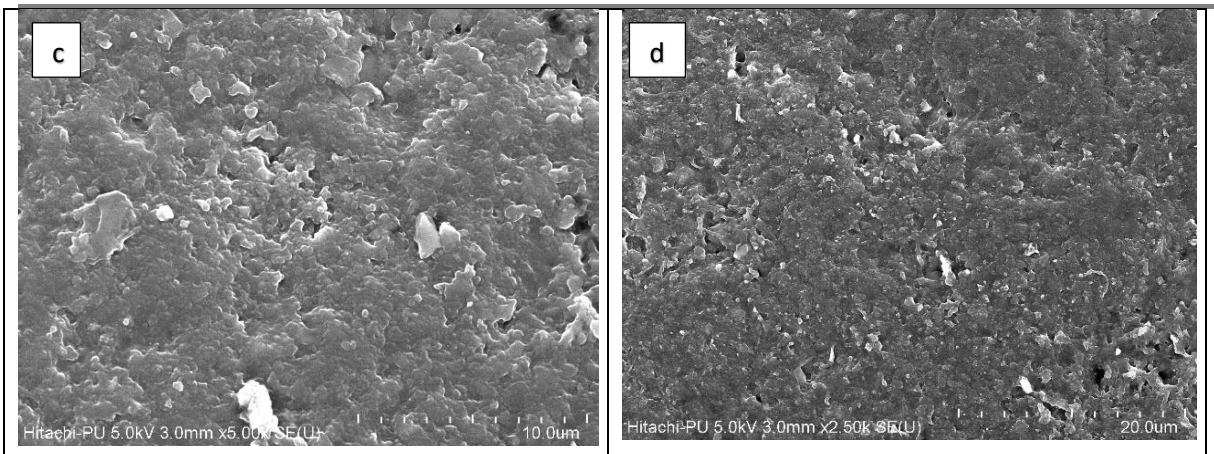


Figure 3.6. SEM micrographs of crosslinked pectin synthesized with different MBAAm concentrations: (a, b) Pectin-cl-MBAAm-1% and (c, d) Pectin-cl-MBAAm-5%, showing irregular, heterogeneous, an d rough surface morphologies at different magnifications.

CONCLUSION

In the present study, crosslinked pectin hydrogels were successfully synthesized by free-radical polymerization using ammonium persulfate (APS) as the initiator and *N,N*-methylene bisacrylamide (MBAAm) as the crosslinking agent. Chemical crosslinking produced stable three-dimensional polymeric networks that exhibited improved structural integrity and water absorption characteristics compared with native pectin. The swelling studies demonstrated that both crosslinked formulations possessed significant water uptake capacity, with the 5% MBAAm-crosslinked polymer exhibiting the highest swelling percentage, indicating the formation of a stable hydrophilic network suitable for aqueous applications. The synthesized crosslinked pectin materials exhibited enhanced aluminium adsorption compared with native pectin. Aluminium removal increased with contact time and was further improved at elevated temperature, demonstrating that the adsorption behaviour was influenced by both structural modification and experimental conditions. The superior adsorption performance of the crosslinked polymer is attributed to the combined effects of improved network stability, increased porosity, enhanced swelling behaviour, and greater accessibility of oxygen-containing functional groups responsible for aluminium binding. FTIR analysis confirmed the successful modification of pectin and indicated the participation of hydroxyl and carboxyl functional groups during aluminium adsorption. SEM observations further revealed a rough and porous surface morphology, which is favourable for water penetration and diffusion of aluminium ions into the polymer matrix. The combined characterization results support the role of chemical crosslinking in improving the physicochemical properties of pectin for adsorption applications. Although the adsorption efficiency obtained in the present investigation demonstrates the potential of crosslinked pectin as a biodegradable and environmentally friendly adsorbent for aluminium removal, the study represents a preliminary evaluation. Overall, the findings demonstrate that MBAAm-crosslinked pectin is a promising natural polymer-based adsorbent with improved structural stability and adsorption characteristics. The study provides a useful foundation for the future development of pectin-derived hydrogel adsorbents for environmental remediation and sustainable water purification technologies.

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