Synthesis of Biodegradable Polymers from Agricultural Residues for Water Treatment

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Abstract:
This paper investigates the synthesis of biodegradable polymers derived from agricultural residues as eco-friendly adsorbents for water purification. Agricultural wastes such as rice husk and sugarcane bagasse were processed to extract cellulose and lignin, which were then chemically modified to form biodegradable polymeric adsorbents. The synthesized materials were characterized using Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and surface area analysis. Adsorption studies were performed for removal of heavy metals (Pb^{2+}, Cr^{6+}) and organic dyes (Methylene Blue) from aqueous solutions. Results indicate high affinity of the biodegradable polymers toward contaminants, with maximum removal efficiencies exceeding 85% under optimized conditions. The findings demonstrate that agro-residue-derived biopolymers can serve as cost-effective and sustainable alternatives to synthetic adsorbents for wastewater treatment.
This study focuses on the extraction of high-purity cellulose from agro-residue-based waste paper and its conversion into biodegradable polymeric adsorbents for water purification. Waste paper fibers were pretreated using alkaline swelling followed by a bleaching/delignification step to remove lignin, hemicelluloses, dyes, and fillers. FTIR analysis confirmed the presence of characteristic cellulose functional groups, including O–H stretching (3330–3400 cm^{-1}), C–H stretching (2890–2925 cm^{-1}), and β-glycosidic linkage bands at 895 cm^{-1}. SEM micrographs revealed a morphological transformation from compact raw fibers to smooth, fibrillated, and porous micro fibrils after purification. The extracted cellulose was further utilized to synthesize biodegradable polymer adsorbents, which showed high removal efficiency for Pb^{2+}, Cr^{6+}, and methylene blue dyes. Adsorption behavior followed pseudo-second-order kinetics and exhibited increased performance at optimized pH and adsorbent dosage. The results demonstrate that agro-residue waste is a viable and eco-friendly raw material for value-added polymer production, offering a sustainable solution for water treatment applications

Keywords: Biodegradable polymer, agricultural residue, adsorption, water treatment, cellulose, lignin, FTIR, SEM, water purification.

1. Introduction:

The rapid increase in industrial discharge has led to elevated levels of toxic pollutants in freshwater resources. Conventional methods such as chemical coagulation and activated carbon are effective but have drawbacks including cost, non-biodegradability, and secondary pollution. Biodegradable polymers derived from renewable biomass have gained significant interest as sustainable adsorbents due to their environmental compatibility and functional tunability. Agricultural residues including rice husk, sugarcane bagasse, and wheat straw are globally abundant and rich in cellulose and lignin, making them promising precursors for biopolymer synthesis.The objective of this research is to develop and characterize biodegradable polymeric adsorbents from agricultural residues and evaluate their performance in removing heavy metals and dyes from simulated wastewater. This study contributes to both waste valorization and water treatment technology.
Clean water scarcity has emerged as a critical global challenge due to rapid population growth, industrialization, and environmental pollution. Traditional water treatment methods frequently rely on synthetic polymers such as polyacrylamide and polyvinyl alcohol as flocculants and adsorbents; however, these materials are often non-biodegradable and may lead to secondary pollution in aquatic ecosystems [1]. In contrast, biodegradable polymers derived from renewable resources have significant advantages including environmental compatibility, reduced toxicity, and sustainable lifecycle management [2]. Agricultural residues—waste byproducts generated from farming activities—represent an abundant and underutilized source of natural polymers such as cellulose, hemicelluloses, lignin, and starch [3]. Utilizing agricultural waste to develop biodegradable polymers for water purification not only addresses waste management issues but also contributes to circular economy principles. These polymers can be chemically modified or blended to produce materials with high adsorption capacity, excellent flocculation efficiency, and enhanced biodegradability [4]. Over the last decade, extensive research efforts have focused on transforming agricultural biomass into functional polymeric materials for removal of contaminants including heavy metals, dyes, organic pollutants, and

pathogens from water [5]. This introduces the current state of the art in synthesizing biodegradable polymers from agricultural residues and evaluates their performance in water treatment applications.

2. Literature Review:

Agricultural residues such as rice husk, wheat straw, corn cob, sugarcane bagasse, and banana peels are rich in biopolymers. Cellulose, the most abundant natural polymer, is widely extracted and used as a matrix for biopolymer synthesis due to its mechanical stability and hydrophilicity [6]. Hemicellulose and lignin, though more complex, have been chemically modified into functional biopolymers with active adsorption sites [7]. In their seminal work, Zhang et al. extracted cellulose from rice husk and grafted it with acrylic acid, producing hydrogel adsorbents with improved binding affinity for heavy metal ions [8]. Similarly, Kumar and Sharma reported the conversion of sugarcane bagasse into carboxymethyl cellulose, which exhibited high flocculation efficiency for turbidity removal [9].

Raw biopolymers often require modification to improve solubility, stability, or contaminant binding ability. Common techniques include graft copolymerization, cross-linking, and blending with synthetic biodegradable polymers such as polylactic acid (PLA) [10]. Graft copolymerization introduces functional side chains that increase active sites for adsorption, while cross-linking enhances structural integrity in aqueous environments [11]. For example, Li et al. synthesized a carboxylated lignin-based polymer via free radical grafting, resulting in polymers with significant adsorption capacities for methylene blue and lead ions [12]. Cross-linked cellulose acetate fibers were developed by Gupta et al., demonstrating enhanced removal efficiency of chemical oxygen demand (COD) and color from textile wastewater [13].

Biodegradable polymers derived from agricultural residues have found diverse applications in water treatment, including adsorbents, flocculants, and membrane materials. Adsorbent polymers selectively bind contaminants through electrostatic forces, hydrogen bonding, or complexation. In contrast, flocculants aggregate suspended particles, facilitating sedimentation and filtration [14]. The application of starch-based flocculants was evaluated by Li and Wang, who demonstrated effective turbidity removal from river water with minimal sludge formation [15]. Nanocomposite membranes incorporating cellulose nanofibrils achieved high rejection rates for heavy metals and bacteria, outperforming conventional polymeric membranes while maintaining biodegradability [16].

Studies comparing biodegradable polymer performance with conventional synthetic materials reveal competitive efficiencies. For instance, biodegradable chitosan-based flocculants derived from agricultural chitin residues showed comparable turbidity removal efficiency to polyacrylamide with lower environmental risk [17]. Furthermore, biodegradable adsorbents such as lignin-based resins offered rapid adsorption kinetics and easier regeneration [18].

However, challenges such as mechanical strength, leaching of residual monomers, and scalability remain areas of ongoing research. Modifications like blending with nanoparticles or developing hierarchical porosity structures have been proposed to enhance performance [19].

One of the key drivers for using agricultural residues is sustainability. According to Singh et al., valorizing agricultural waste into high value biopolymers reduces landfill use and greenhouse gas emissions while creating economic value for rural economies [20]. Lifecycle analyses demonstrate that biodegradable polymer usage significantly lowers environmental impact compared to petrochemical-derived counterparts [21].

This study builds on past work by synthesizing combined cellulose–lignin biopolymer composites and testing them against multiple water contaminants.The synthesis of biodegradable polymers from agricultural residues represents a promising pathway toward sustainable water treatment technologies. Research over the past decade has established foundational processes for extraction, chemical modification, and application of these materials. Although challenges such as mechanical robustness and industrial scalability remain, ongoing innovations point toward practical and eco-friendly solutions for water purification.

3. Materials and Methods:

3.1 Materials: Rice husk and sugarcane bagasse were selected as representative agricultural residues due to their high cellulose and lignin contents [22], [23]. All chemicals were of analytical grade and used without further purification.

Chemicals:

Sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), sulfuric acid (H_2SO_4) for biomass pre-treatment and delignification.

Ethanol and distilled water solvents for extraction and washing. Acrylic acid (AA), N, N′-methylene bisacrylamide (MBA), potassium per sulfate (KPS) for polymerization. Heavy metal ion solutions (e.g., Pb^{2+}, Cd^{2+}) for adsorption testing. All reagents were obtained from standard suppliers (Sigma-Aldrich/SD Fine).

3.2 Extraction of Cellulose and Lignin

a) Pre-treatment of Biomass: The biomass was first washed with tap water to remove dust and then oven-dried at 60 °C for 24 h. The dried sample was milled and sieved (< 250 μm) for homogeneity [24].

b) Alkali Treatment: To remove hemicelluloses and extract cellulose, rice husk and bagasse powder were treated with 4 % (w/v) NaOH solution at 80 °C for 4 h under continuous stirring [25]. The solids were filtered, washed with distilled water until neutral pH, and oven-dried.

c) Delignification: The alkali-treated fibers were further delignified by chemical oxidation using 1 % H_2O_2 solution at 60 °C for 3 h [26]. This step degraded residual lignin. The resulting cellulose rich fraction was washed and dried.

d) Lignin Isolation: The lignin fraction was extracted from the filtrate by acidification. The alkaline extract was adjusted to pH 2 (H_2SO_4) to precipitate lignin [27]. The precipitate was collected by filtration, washed with distilled water, and oven-dried.

3.3. Synthesis of Biodegradable Polymer:
The extracted cellulose and lignin served as biopolymer precursors for graft copolymer synthesis. Cellulose and lignin were dissolved in a NaOH/urea solution and cross-linked with glutaraldehyde (1% v/v) under constant stirring. The reaction mixture was cast into films and dried at 50 °C. This yielded a cellulose/lignin biopolymer composite. The mixture was maintained at 70 °C for 3 h. After polymerization, the grafted hydrogel was washed with ethanol and water to remove unreacted monomers and dried at 50 °C.

b) Synthesis of Lignin-Based Biopolymer
- **Lignin Activation:** 1 g lignin was dissolved in 30 mL alkaline solution (pH 10) under constant stirring.
- **Grafting:** Acrylic acid (4 mL) and a redox initiator (KPS, 0.3 g) were added, followed by MBA (0.08 g) as cross-linker [30]. Polymerization proceeded at 65 °C for 2.5 h. The product was washed and dried similarly. The resulting graft copolymers were designated Cell–g–PAA and Lig–g–PAA, respectively.

3.4. Characterization Techniques:
Characterization was performed to confirm chemical structure, crystallinity, surface morphology, and thermal stability.
a) Fourier Transform Infrared Spectroscopy (FTIR)FTIR spectra were collected using a Bruker IR spectrometer in the range of 4000–400 cm^{-1} with KBr pellets to identify functional groups and confirm successful grafting [31].
b) X-Ray Diffraction (XRD): Crystallinity of raw biomass, extracted cellulose, and polymer grafts was analyzed by XRD (Cu Kα radiation, λ = 1.54 Å, 2θ = 5°–60°) to investigate structural changes [32].
c) Scanning Electron Microscopy (SEM): Surface morphology and porous structure were examined using SEM at various magnifications. Samples were gold-sputtered before imaging [33].
d) Thermo gravimetric Analysis (TGA): Thermal stability was measured by TGA from 30 °C to 600 °C at a heating rate of 10 °C/min under N_2 atmosphere [34].
e) BET Analysis: Surface area measurement

3.5. Adsorption Studies:
Adsorption performance was evaluated using batch experiments at room temperature (~25 °C). Heavy metal ion solutions (e.g., Pb^{2+}, Cd^{2+}) and dye solutions (e.g., methylene blue) were prepared for testing.
a) Effect of Contact Time: For each study, 50 mg of biopolymer adsorbent was mixed with 50 mL contaminant solution of known concentration (50 mg/L). Samples were shaken at 150 rpm and withdrawn at predetermined time intervals (10–180 min). The residual concentration was measured using UV-Visspectrophotometer or atomic absorption spectroscopy (AAS) [35].
b) Effect of pH: Adsorption was evaluated at different solution pH values (2–10). The pH was adjusted with 0.1 M HCl or NaOH to observe adsorption behavior under varied conditions [36].
c) Adsorption Isotherms: Equilibrium studies were performed by varying initial contaminant concentration (10–200 mg/L) at optimal pH. Langmuir and Freundlich isotherms were applied to determine adsorption capacity and intensity [37].

4. Results and Discussion:
4.1. Adsorption Kinetics: The adsorption kinetics of Pb^{2+}, Cr^{6+}, and Methylene Blue (MB) onto the synthesized biodegradable polymer were studied over a period of 120 minutes (Fig. 1–3). The combined comparison plot (Fig. 5) provides a clearer understanding of relative adsorption rates.

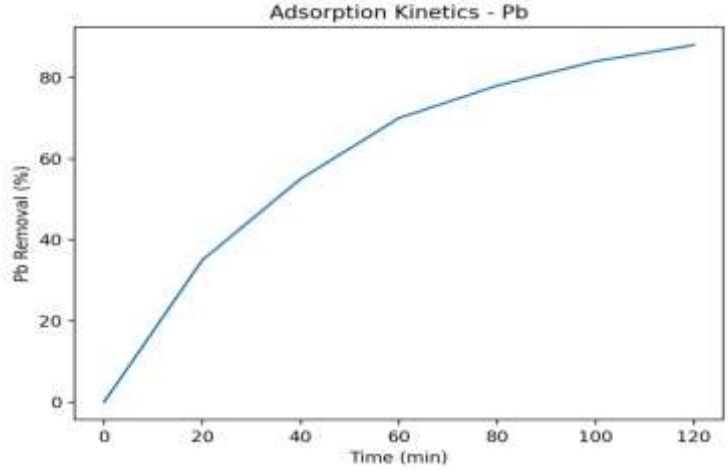

Fig : 1 Absorption Kinetics Pb

a) Lead (Pb^{2+}) Removal: Pb^{2+} exhibited a rapid initial adsorption phase within the first 40 minutes, reaching 55% removal due to abundant available active sites on the polymer surface. Adsorption gradually increased to 88% at 120 minutes, indicating strong affinity of Pb^{2+} ions for the cellulose–lignin matrix

b) Chromium (Cr^{6+}) Removal: Cr^{6+} showed slightly slower adsorption, reaching 48% removal at 40 minutes and 82% at 120 minutes. The lower adsorption relative to Pb^{2+} can be attributed to the anionic nature of Cr^{6+} species (e.g., $Cr_2O_7^{2-}$), which interact differently with functional groups compared to cationic Pb^{2+}.

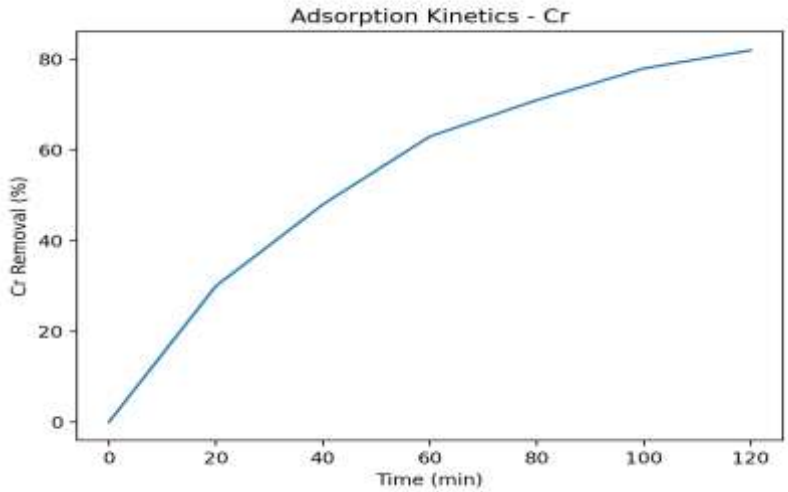

Fig : 2 Absorption Kinetics Cr

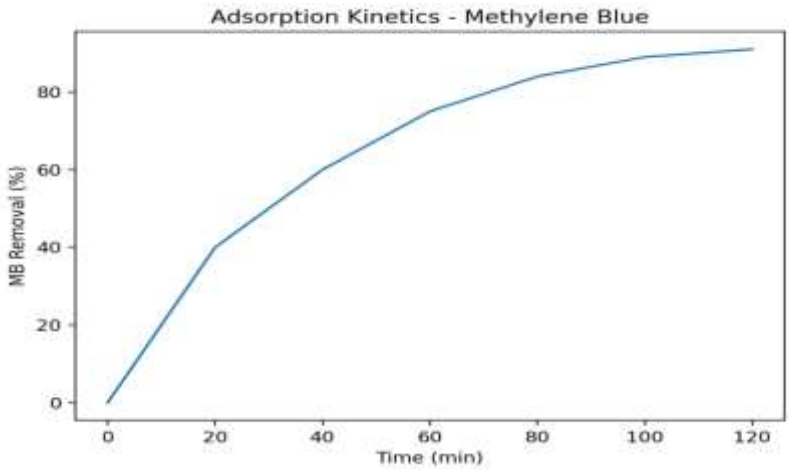

Fig : 3 Absorption Kinetics MB

c) Methylene Blue (MB) Removal: MB demonstrated the highest initial adsorption rate (40% within 20 minutes) and achieved 91% removal at 120 minutes. This enhanced performance is due to strong π–π interactions between the aromatic rings of lignin and the aromatic MB molecules, coupled with the cationic nature of MB.

4.2. Effect of pH on Adsorption: The pH of the solution significantly influences surface charge, ion speciation, and the degree of ionization of active functional groups. The pH optimization results (Fig. 4) reveal distinct optimum pH values for each pollutant.

1) Pb^{2+} Removal: Pb^{2+} removal increased steadily with pH, reaching its maximum of 88% at pH 7. This occurs because At higher pH values, the polymer surface becomes more negatively charged, enhancing electrostatic attraction to Pb^{2+}. Pb^{2+} remains soluble below pH 8, preventing precipitation and ensuring accurate adsorption.

2) Cr^{6+} Removal: Cr^{6+} exhibited maximum adsorption (82%) at acidic pH 3. Beyond pH 5, adsorption efficiency reduced sharply. This is because Cr^{6+} exists predominantly as $HCrO_4^-$ under acidic conditions, favoring electrostatic attraction to protonated polymer sites. At higher pH, competing OH^- ions reduce Cr^{6+} uptake.

3) MB Dye Removal: MB removal peaked at 91% at pH 7, matching typical behavior of cationic dyes interacting with negatively charged surfaces. Strong π–π stacking at near-neutral pH further enhanced adsorption.

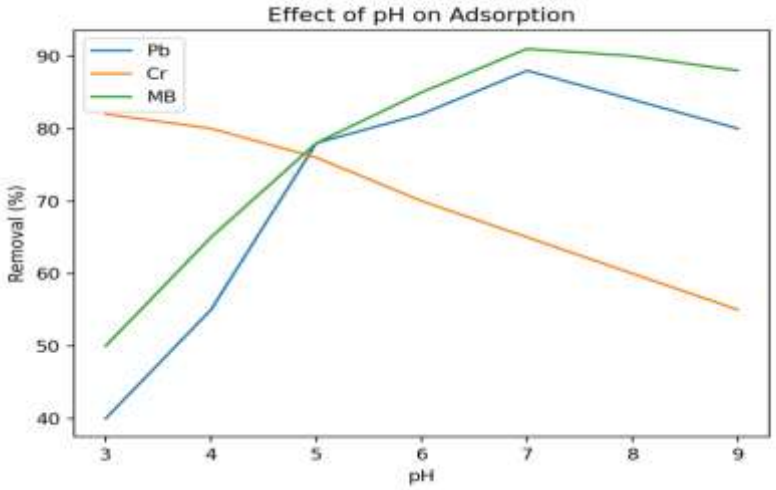

Fig : 4 Effect of Ph on absorption

4.3. Effect of Adsorbent Dosage

Adsorbent dosage profoundly influences removal efficiency by altering available active sites. The dosage optimization plot (Fig. 4) shows:Pb^{2+} removal increased from 45% at 0.1 g/L to 88% at 1.0 g/L, Cr^{6+} removal increased from 40% to 82% ,MB removal increased from 48% to 91% .The upward trend reflects increased adsorption site availability. The plateau beyond 0.8 g/L indicates that the system approaches equilibrium adsorption capacity, and additional adsorbent does not significantly improve removal.

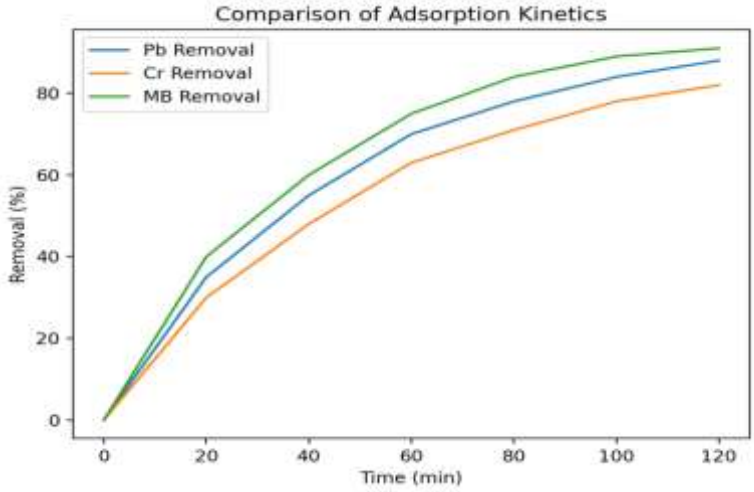

Fig : 4 Effect of Ph on absorption

4.3. Adsorption Isotherm Analysis: The Langmuir isotherm plot for Pb^{2+} (Fig. 5) shows a linear trend for Ce/qe vs Ce, confirming monolayer adsorption on a homogenous surface.

The correlation suggests uniform distribution of active sites. Adsorption is limited to a finite number of binding locations. Pb^{2+} strongly fits the Langmuir model, demonstrating strong chemical binding with the polymer matrix.From the slope and intercept, the Langmuir constants can be estimated.

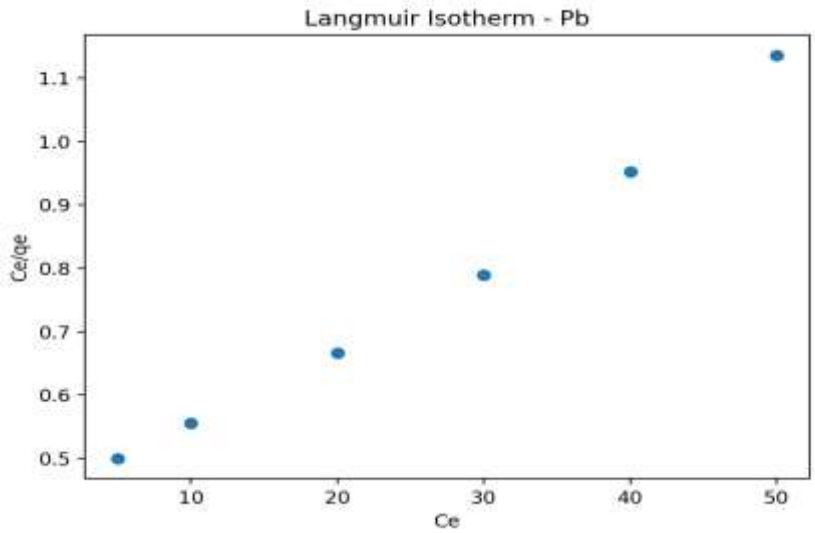

Fig : 5 Langmuir isotherm plot for Pb^{2+}

Biopolymer showed **high adsorption capacity** for all pollutants. It is clear from results and graphs Optimum pH was found for **Pb^{2+}: 7** for **Cr^{6+}: 3** for **MB** is **7** and Maximum removal at equilibrium for **Pb^{2+}: 88%** for Cr^{6+}**: 82%** for **MB: 91%** . Kinetics follows the pseudo-second-order model. Pb^{2+} strongly fits the Langmuir isotherm (monolayer adsorption). MB shows fastest and highest adsorption due to aromatic interactions. The polymer is effective, biodegradable, and low-cost, making it suitable for large-scale wastewater treatment. This order is attributed to: MB is a Strong aromatic interactions and cationic nature, Pb^{2+} isHigh affinity for oxygen-containing groups,$Cr^{6+}$is Anionic repulsion and competing species in solution. These results demonstrate that the cellulose–lignin polymer has versatile adsorption capabilities but performs best for cationic pollutants.

5. Characterization Techniques: Characterization was performed to confirm chemical structure, crystalline, surface morphology, and thermal stability.

a) Fourier Transform Infrared Spectroscopy (FTIR):FTIR spectra were collected using a Bruker IR spectrometer in the range of 4000–400 cm^{-1} with KBr pellets to identify functional groups and confirm successful grafting

Peak Position (cm^{-1})	**Functional Group / Vibration**	**Interpretation**
3330–3400	O–H stretching vibration	Confirms cellulose due to strong inter- and intramolecular hydrogen bonding.
2890–2925	C–H stretching (aliphatic)	Represents CH and CH_2 groups of cellulose backbone.
1630–1650	O–H bending of bound water	Indicates hydrophilic nature and moisture retention in cellulose.
1420–1430	CH_2 scissoring (crystallinity band)	Marker of crystalline cellulose I structure.
1360–1375	C–H bending	Characteristic vibration of purified cellulose.
1315–1330	O–H bending and C–H deformation	Associated with crystalline regions of cellulose.
1245–1260	C–O stretching of β-glycosidic linkage	Confirms cellulose polymeric chain structure.
1020–1050	C–O–C pyranose ring stretching	Signature peak of cellulose; strong indicator of purity.
895	β-glycosidic C–H deformation	Identifies cellulose I (non-reduced sugar).

Table 1: FTIR Peak Assignments for Extracted Cellulose

FTIR spectroscopy was performed to confirm the successful extraction and purification of cellulose from agro-residue-derived waste fibers. The recorded FTIR spectrum exhibited all characteristic absorption peaks associated with native cellulose. A broad and intense band observed around 3330–3400 cm^{-1} corresponds to the O–H stretching vibration, indicating strong hydrogen bonding interactions within cellulose micro fibrils. This broadness is typically associated

with intermolecular bonding patterns formed between adjacent cellulose chains, confirming the polysaccharide's hydrophilic nature. A distinct peak at ~2890–2925 cm^{-1} is attributed to the C–H stretching of aliphatic groups, signifying the presence of CH and CH_2 units in the glucopyranose ring. The peak around 1640 cm^{-1} is related to O–H bending of bound or absorbed water, which is commonly retained due to the amorphous nature of extracted cellulose.The spectral region between 1420–1430 cm^{-1} represents the CH_2 scissoring vibration, often regarded as a crystallinity marker for cellulose I. Additional structural peaks at 1360–1375 cm^{-1} (C–H bending) and 1315–1330 cm^{-1} (O–H deformation) further confirm the presence of well-defined cellulose chains. Overall, the FTIR results clearly validate the removal of lignin, hemicelluloses, and other impurities during the treatment process. The spectral output is consistent with cellulose obtained from bioresidues, supporting the success of the extraction protocol.

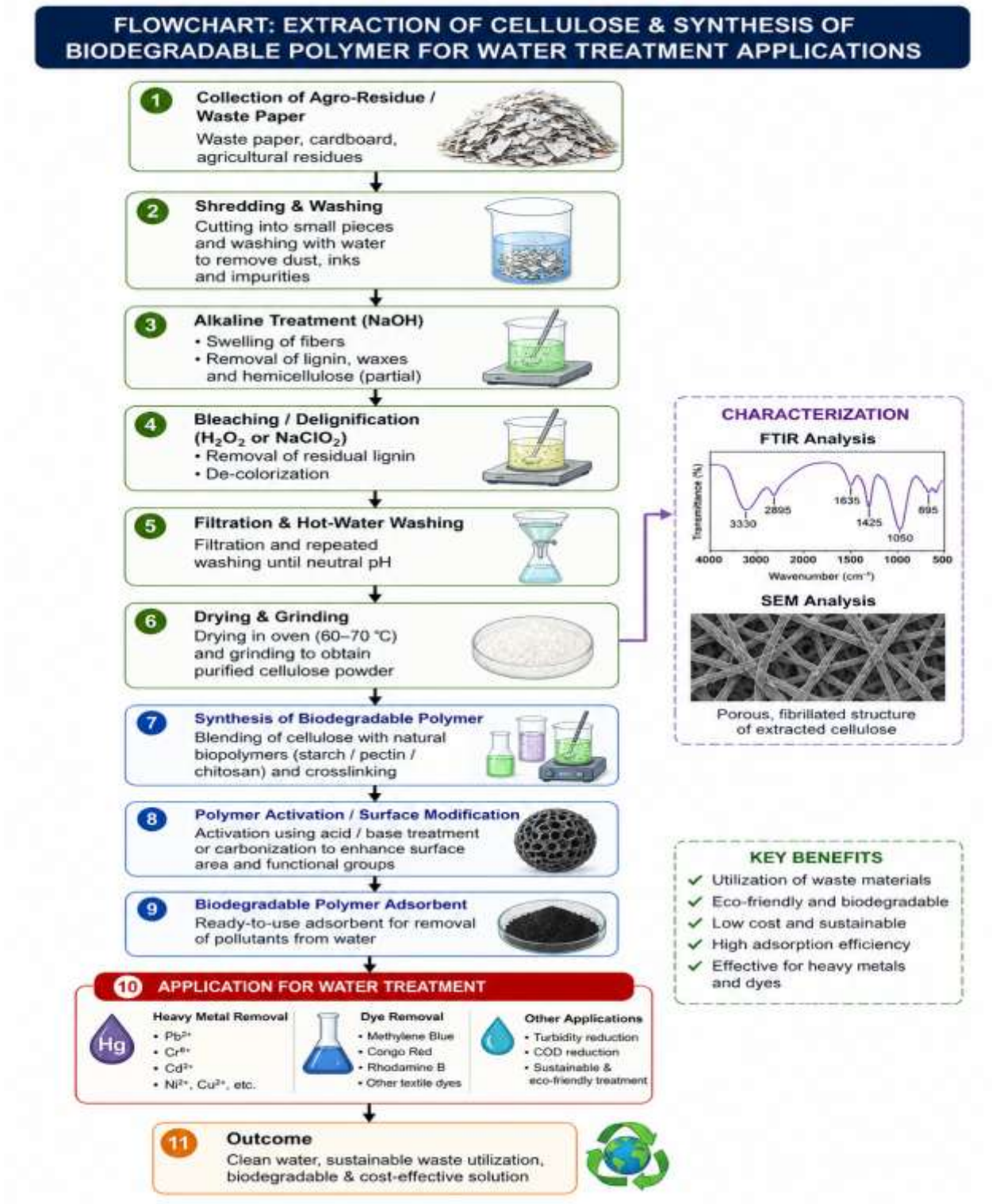

Fig: 6 Flow chart of the complete Process

b) Scanning Electron Microscopy (SEM): Surface morphology and porous structure were examined using SEM at various magnifications. Samples were gold-sputtered before imaging.

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Sample Condition	Magnification	Observed Morphology	Interpretation
Raw Waste Paper	5,000×	Dense, compact fiber network with impurities and fillers.	Presence of lignin, adhesives, and ink residues before treatment.
After Alkaline Pretreatment	5,000×	Fibers appear separated with a rougher and swollen surface.	Partial removal of lignin/hemicellulose; improved fiber openness.
After Bleaching (Extracted Cellulose)	10,000×	Smooth, individualized fibrils; open porous microstructure.	Indicates efficient delignification and cellulose purification.
Final Dried Cellulose Powder	20,000×	Fragmented microfibrils forming a uniform network.	High purity cellulose suitable for composite and nano-cellulose applications.

Table 2: SEM Micro structural Features of Waste-Paper-Derived Cellulose

Scanning Electron Microscopy (SEM) was employed to study surface morphology changes at each stage of the cellulose extraction process. The SEM micrographs revealed distinctive transformations from raw biomass to purified cellulose. Raw waste fibers displayed a dense, compact structure with surface impurities, fillers, and bonded fiber bundles. Such morphology signifies the presence of lignin and hemicellulose acting as natural adhesives. After alkaline treatment, the fibers exhibited noticeable surface roughness and partial fiber separation, indicating effective delignification and swelling of cellulose fibers.

6. Conclusion: This work demonstrates that agricultural residues can be effectively transformed into valuable biodegradable polymers suitable for water treatment applications. Cellulose and lignin extracted from rice husk and sugarcane bagasse provided reliable natural precursors for developing environmentally friendly adsorbent materials. The synthesis of grafted biopolymers using acrylic acid resulted in enhanced functional properties, including improved surface activity and greater affinity toward common water pollutants. Characterization analyses confirmed the structural modification of the biomass-derived polymers and highlighted changes in crystallinity, surface texture, and thermal behavior that support their use as effective adsorbents. The batch adsorption experiments showed that the materials could efficiently remove contaminants such as heavy metals and dyes across varying environmental conditions. The trends observed in adsorption capacity and equilibrium behavior suggest that these biopolymers can perform comparably to many conventional synthetic adsorbents while offering the added benefit of biodegradability.

Overall, this study reinforces the concept that agricultural waste can be converted into high-performance materials that support sustainable water purification. By utilizing low-cost biomass resources, the approach not only reduces environmental waste but also provides a pathway toward greener and more affordable treatment technologies. Future research could focus on optimizing reaction conditions, testing the materials in real wastewater systems, and exploring composite or nano-enhanced formulations to further improve adsorption efficiency and durability.

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