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Review

# Production of functionalized clay-CNC based biopolymeric nanocomposite from agro-waste biomass for bulky industrial wastewater treatment via continuous column adsorption study with mathematical modeling: A critical review

Md. Mahmudur Rahman [a,b,*], Songita Rani Adhikary [b], M Mohinur Rahman Rabby [d], Md. Mahafujul Hassan [d], Salah Knani [c], Syed Hasibul Akhfer [b,e], Md. Khalid Al Zuhanee [d]

[a] BCSIR, Rajshahi Laboratory, Bangladesh Council of Scientific and Industrial Research (BCSIR), Rajshahi, 6206, Bangladesh
[b] Department of Applied Chemistry & Chemical Engineering, Islamic University, Kushtia, 7003, Bangladesh
[c] Center for Scientific Research and Entrepreneurship, Northern Border University, Arar, 73213, Saudi Arabia
[d] Department of Chemical Engineering, Rajshahi University of Engineering and Technology (RUET), Rajshahi, 6204, Bangladesh
[e] Human Resource Development (HRD), Gazipur Digital University, Kaliakair, Gazipur, 1750, Bangladesh

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ABSTRACT

Due to extensive industrialization, fluctuating technologies, and an overgrowing population a colossal amount of hazardous effluent is generated every moment and discharged directly into the environment. This crude wastewater can contaminate the environment and food cycle; causing serious damage not only to the ecosystem but also to public health. Therefore, it is critical to treat this wastewater by improving an ecological and economical technology before discharging/reusing. However, researchers are exasperating to improve an efficient and effective technology for the refinement of the bulky industrial wastewater. Here, some state-of-the-art production techniques of the FC-CNC bionanosorbents have been proposed for the effective purification of bulky effluent via a continuous adsorption system. This technology is superior to other methods due to its lower cost, higher efficiency, innovative and beneficial nature, and high selectivity. Instead, the precursors (cellulose and clay) are more available, nontoxic, ecofriendly, biodegradable, thermally and mechanically stable, and have shown high surface area, porous structure, possessed substantial active binding sites that could have been busting their adsorption performances. Still there are several complications concerning the effective processing/modification of the main precursors, production techniques, mechanism, and mode of application while purifying the crude wastewater. Henceforth, this review paper would be devoted by concentrating the modification process, advance production methods, synergistic mechanism to support the mono and/or multilayer physisorption, chemisorption, diffusion/deep penetration, and mode of application addressing the bulky wastewater treatment. By discovering the gaps and suggesting impending instructions, this study aims to expand the fabrication, categorization, and application of the FC-CNC bionanoadsorbents.

## 1. Introduction

Global industrialization has profoundly transformed human existence by augmenting economic prosperity, elevating living standards, and catalyzing technological innovations. Concurrently, this ongoing wave of industrialization and globalization is giving rise to pervasive threats of air, water, and soil pollution on a global scale (Rahman et al., 2024a). Evidently, water contamination has upraised grave concerns to humanity and exacerbated perilous health-related issues. There is a huge depletion of adequate clean water to fill drinking water demands and sanitation requirements, thereby impeding human health and productivity. Approximately 780 million individuals around the world remain devoid of access to safe drinking water sources (Rahman et al., 2025b). The global shortage of potable water profoundly affects human life, particularly in underdeveloped nations and vulnerable populations. Approximately 36 % of the global population faces water stress, with

* Corresponding author. BCSIR, Rajshahi Laboratory, Bangladesh Council of Scientific and Industrial Research (BCSIR), Rajshahi, 6206, Bangladesh.
*E-mail address:* shamrat.acce@gmail.com (Md.M. Rahman).

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over two billion people using contaminated water. Water pollution drives ecological decline and biodiversity loss. By 2030, water scarcity is expected to displace over 700 million people, and by 2050, it will threaten one billion livelihoods across 100 countries. Despite these challenges, societal efforts to conserve and protect water remain woefully inadequate (Goh et al., 2016). Industrial wastewaters often act as a reservoir for a myriad of hazardous pollutants, encompassing both organic and inorganic dyes, toxic heavy metals, pharmaceutical residues, medical wastes, and fragments of micro and nanoplastics (Rahman, 2024; Sheikh et al., 2023a,b; Sundararaman et al., 2025). Moreover, research indicates that wastewater harbors approximately 700 micropollutants-both organic and inorganic, many of which are profoundly toxic and carcinogenic (Iqbal, 2016; Rahman and Manir-uzzaman, 2021). A significant number of these substances are neither biodegradable nor subject to biotransformation, while others exhibit extended persistence in the environment (Unuabonah and Taubert,

**Table 1**
A list of hazardous toxic pollutants, their source of discharge, their bad effect on human health and environment, as well as their permissible limit along with references.

| Toxicants | Source | Health Effects | Maximum allowance | References |
|---|---|---|---|---|
| Arsenic (As) | Originates from fertilizers, chemical production, oil refineries, and pesticide industries. | Prolongs the risk of skin cancer, lung and kidney damage, and other internal malignancies. It also increases infant mortality, reduces birth weight, and causes neurobehavioral disorders and genotoxic effects. | 0.050 mg/L | Abbas et al. (2016); Barakat (2011); Ojedokun and Bello (2016); Tran et al. (2017). |
| Lead (Pb) | Originates from chemicals production, distilleries, dye manufacturing, fertilizers industry, food industry, household waste, metal smelting, pharmaceuticals industry, sugar industry, and wastewater sludge. | Results in kidney destruction, heart failure, liver damage, skin and eye irritation, impaired hemoglobin synthesis, central nervous system damage, and cognitive impairment. It can also lead to blood diseases like anemia and hypertension. | 0.050 mg/L | Rahman et al. (2023d); Gilani et al. (2015); Rahman et al. (2022); Carolin et al. (2017); Qu et al. (2013); Flora et al. (2012); Sharma and Agrawal (2005). |
| Cadmium (Cd) | Originates from engineering industry, coal burning, dairy industry, distillery, electroplating process, metal smelting, and petroleum industry. | Seriously affects the kidneys, leading to rapid damage, bone pain, liver syndromes, hypertension, and a substantial risk of cancer development. It also causes nausea, vomiting, diarrhea, headaches, and osteoporosis. | 0.005 mg/L | Nordberg et al. (2018); Suhani et al. (2021); Bhattacharyya et al. (2023); Barakat (2011). |
| Chromium (Cr) | Associated with the tanning industry, dye manufacturing, aircraft manufacturing, fine chemicals industry, and distilleries. | Induces nausea, diarrhea, lung obstruction, liver and kidney impairment, cancer, skin disorders, nervous and digestive system issues, and thyroid malignancies | 0.050 mg/L | Hu et al. (2009); Lokhande et al. (2011); Kharayat (2012); Chowdhury et al. (2015). |
| Cobalt (Co) | Linked to steel manufacturing, mining zones, and petroleum refining industries. | Affects the respiratory system and skin, potentially leading to hypersensitivity lung disease and dermatitis. It also impacts the heart, thyroid, liver, and kidneys. | 0.010 mg/L | Leyssens et al. (2017); Tran et al. (2017); Paustenbach et al. (2013); Dunlop et al. (2005); Jadaa (2024). |
| Nickel (Ni) | Found in paper mills, electrochemical industries, garment and textile mills, dyeing and paint making, fertilizers industry, and petroleum combustion. | Causes asthma, chronic lung disease, dry cough, nasal congestion, bluish skin, chest tightness, rapid breathing, breathlessness, dizziness, skin allergies, and pulmonary illnesses. | 0.070 mg/L | Rahman et al. (2023a), 2024a; Yang et al. (2009); Das et al. (2018); Abbas et al. (2016); Sheikh et al., 2023a,b. |
| Manganese (Mn) | Associated with soap and detergents, blast furnaces, and paper mills. | Affects the central nervous system, potentially leading to alterations in neurological and neurobehavioral functions. Symptoms include mood changes, reduced motor skills, limb numbness, and diminished memory. | 0.020 mg/L | Michalke and Fernsebner (2014); O'Neal and Zheng (2015); Crossgrove and Zheng (2004); Kowalska et al. (2005). |
| Copper (Cu) | Associated with dye and electronic device manufacturing, aircraft manufacturing, chemicals production, dairy industry, engineering industry, and pesticides industry. | Causes hypertension, sleeplessness, rapid respiration, seizures, muscular cramps, skin and brain damage, cancer progression, angiogenesis, inflammation, neurological disorders, diabetes, cardiovascular disease, atherosclerosis, liver diseases, anemia, intestinal problems, and long-term kidney and liver harm. | 0.020 mg/L | Rahman et al. (2023a); Ebrahimi et al; Hoang et al. (2022); Chan et al. (2010); Afolabi et al. (2016); Alluri et al. (2007); El Messaoudi et al., 2024a,b. |
| Mercury (Hg) | Linked to pesticides industry, electrolysis processes, fertilizers industry, organic chemistry, plastic manufacturing, and steel manufacturing. | Impacts the neurological system, causing central neural system damage and nephrotoxic effects. It also affects the brain and kidneys, and various physical systems like immune and respiratory systems. | 0.001 mg/L | Bridges and Zalups (2017); Yaseen and Scholz (2019); El Messaoudi et al., 2024a,b; Alluri et al. (2007); Tran et al. (2017); Fernandes Azevedo et al., 2012. |
| Zinc (Zn) | Linked to pharmaceuticals industry, blast furnaces, chemicals production, dairy industry, electroplating process, soap and detergents. | Associated with fatigue, increased thirst, stomach sickness, depression, nervousness, muscular cramps, skin inflammation, and vomiting. | 0.80 mg/L | Hoang et al. (2022); Abbas et al. (2016); Plum et al. (2010); Chasapis et al. (2012); Alluri et al. (2007). |
| Dyes | Originates from garments/textile mills, dyeing/paint making. | Causes kidney destruction, heart failure, liver damage, skin and eye irritation, central nervous system damage, and brain damage. | >0.00001 mg/L | Khan et al. (2023); Rahman et al. (2024d); Pang et al. (2021); Dutta et al. (2024). |
| Antibiotic residue | Found in livestock and poultry products, aquatic products, hospital waste. | Can cause cancers, allergic reactions, chronic toxic effects, disrupt the digestive system, and lead to childhood obesity. | >0.0000082 mg/L | Menkem et al. (2019); Danner et al. (2019); Wang et al. (2023); Ben et al. (2019); Arsène et al., 2022; Chen et al. (2019). |
| Pesticides residue | Associated with agriculture and farming sectors. | Leads to stinging eyes, rashes, blisters, blindness, nausea, dizziness, diarrhea, death, cancers, birth defects, reproductive harm, immunotoxicity, neurological and developmental toxicity, and disruption of the endocrine system. | >0.0005 mg/L | Shukla et al. (2022); El-Nahhal and El-Nahhal, 2021; Taiwo (2019). |

2014). Toxic heavy metals are perceived as prevalent and deleterious contaminants. Their propensity to transform into nanoforms facilitates water matrix pollution, and their diminutive size enables them to infiltrate semipermeable membranes of plants and aquatic organisms. Consequently, they can contaminate the food chain, severely compromising human health (Rahman et al., 2022, 2024d). Furthermore, there exists more deleterious organic and inorganic pollutants, including dyes, micro- and nanoplastics, humic substances, pesticides, herbicides, polycyclic aromatic hydrocarbons, phenols, drug residues, chemical residues like inorganic salts, acids, alkalis and so on, severely harm the environment and biological systems (Bhatti et al., 2017; Baskar et al., 2025; Rashid et al., 2020; Ciğeroğlu et al., 2024; Rahman et al., 2023d & 2025a). Their release into aquatic systems disturbs ecological equilibrium, leads to bioaccumulation within food webs, and jeopardizes the safety of drinking water, sanitation, and irrigation practices (Hokkanen et al., 2016; Rahman et al., 2023a) whereas pregnant women and growing child are in the most vulnerable conditions. It is important to mention that these crude industrial wastewater has currently been marked as a breakneck treads not only for the environment but also for the public health safety/security. For better clarity, a list of hazardous toxic pollutants, their source of discharge, their bad effect on human health and environment, as well as their permissible limit along with references has been given below in Table 1. Hence, this problem should be solved as early as possible by developing a cost effective, ecofriendly, and high efficient technology. Meanwhile, a number of conventional technologies have been devised to tackle the pressing issue of wastewater management. Despite their widespread adoption, traditional water treatment methods, including floatation, chemical oxidation-reduction; coagulation-flocculation, (Khan et al., 2016; Szczepanik, 2017),liquid-liquid extraction, membrane filtration, electro-precipitation, (Fito and Nkambule, 2023; Sheikh et al., 2023a,b; Zhang et al., 2017), evaporation, ion exchange, photo-electro catalysis (Zhu et al., 2016), reverse osmosis, irradiation (Deghles and Kurt, 2016), removal (Kumar et al., 2024; Sundararaman et al., 2024) and so on, often prove inadequate for efficacious water treatment due to the significant drawbacks. Such as: some techniques often require costly equipment and can inadvertently generate hazardous byproducts. This inadequacy underscores the need for more advanced and integrated approaches that can effectively address the complex nature of modern wastewater pollutants (Mohammed et al., 2018). The choice of a particular treatment process for wastewater treatment is predominantly influenced by a complex array of factors, encompassing waste type and concentration, effluent heterogeneity, and the necessary level of treatment, in conjunction with financial considerations (Rahman, 2024; Sayyed et al., 2021). Hench, lately considerable emphasis has been placed on the development of state-of-the-art technologies for water and wastewater treatment, among these methods adsorption process is frequently regarded as the optimal method for treating industrial wastewater, also emerging as a particularly efficacious method due to its straightforward design and minimal initial investment, as well as the limited land required (El-Messaoudi et al., 2024 & 2017; Sunkar et al., 2023; Rahman et al., 2022, 2023d & 2025b; Rashid et al., 2017). Additionally, this well-known method provides distinct advantages in its capacity to eliminate both organic in addition to inorganic pollutants from water bodies. This is achieved by leveraging both synthetic and bio-based active polymeric compounds derived from natural sources. Notable examples include cellulose, lignin, cellulose nanocrystals (CNC), chitosan, chitin, activated carbon produced from biomass, charcoal, biochar, and other similar materials (Rahman et al., 2025a). These compounds are integral to the method's effectiveness, as they can be utilized individually or in combination to enhance pollutant removal efficiency. The incorporation of such natural materials not only underscores the method's eco-friendliness but also highlights its potential for cost-effective and sustainable water purification solutions (Rahman and Maniruzzaman, 2019; Rahman, 2024; Ashraf et al., 2024). It has recently been recognized that for the continuous treatment of industrial wastewater on a large scale in real-time, fixed bed column adsorption is more efficacious compared to the batch approach. The batch experimentation has shown certain shortcomings, including the requirement for a fixed volume of wastewater samples, the inappropriate utilization of highly concentrated toxicants in the feed solution, and difficulty recovering used nanocomposites or adsorbents. In contrast, fixed bed column adsorption offers a more efficient and scalable solution, allowing for the continuous processing of wastewater while minimizing the drawbacks inherent to batch systems (in Table 5). This method enables the treatment of a larger volume of wastewater using a defined quantity of adsorbent, making it more suitable for real-world applications where continuous operation is necessary. Additionally, the reuse of adsorbents in column systems can enhance the sustainability and cost-effectiveness of the treatment process (Thirunavukkarasu et al., 2021). The selection of an optimal adsorbent for the purification of wastewater is primarily dictated by the comprehensive chemical characteristics of the wastewater. Key parameters include pH, salinity, conductivity, hardness, and total dissolved solids (TDS), as well as physical properties like viscosity, total suspended solids (TSS), turbidity, and overall solid content. However, surpassing these factors in importance is the nature and concentration of micro and nanopollutants, along with toxicants present in the wastewater. These contaminants significantly influence the treatment strategy and efficacy. Moreover, the adsorptive efficacy of the biosorbent towards its intended adsorbate, as well as a comprehensive efficiency and cost analysis, must be taken into account. These biosorbents may possess several advantageous attributes, including being cost-effective, non-toxic, renewable, porous, and widely accessible. Furthermore, they should exhibit enhanced thermal stability, a substantial specific surface area, a multitude of active sites, optimal surface morphology and crystallinity, and a minimal propensity for generating excessive sludge. Nonetheless, adsorbents derived from organic, inorganic, hybrid, or biological materials have consistently demonstrated unequivocal benefits in the purification of polluted water or wastewater. These including cellulose nanocrystals based nanofilter (Sheikh et al., 2023a,b), activated charcoal (Rahman et al., 2024d; Nam et al., 2014), both natural and modified zeolites ((Aragaw and Ayalew, 2019; Chen et al., 2020), clay minerals and clay based composites (Rahman and Maniruzzaman, 2021; Ayalew, 2020; Thiebault, 2020), biochar (Rahman et al., 2025a; Fito et al., 2022; Premarathna et al., 2019), chitosan based composites (Kumar et al., 2024; Rahman et al., 2024f; Biswas et al., 2020; Rusmin et al., 2015), agro-wastes (Joseph et al., 2021; Sunkar et al., 2023; Rahman and Maniruzzaman, 2021), both natural and modified biomass (Coelho et al., 2020; Kumararaja et al., 2018) can be considered as some notable instances of highly effective adsorbents or biosorbents.

Currently, researchers worldwide are keenly interested in natural clays, which are considered the most popular adsorbents due to their superior adsorption efficiency against toxicants, notable surface morphology, robust crystal structure, antimicrobial properties, non-toxicity, thermal stability, cost-effectiveness, and widespread availability (Rahman, 2024). Nonetheless, the use of immaculate clay minerals is severely restricted due to their inferior capacity to remove micro- and nanopollutants compared to zeolites and activated carbon. These limitations stem from their smaller specific surface area, challenges in recovery and regeneration after adsorption, and low affinity for organic pollutants caused by hydration, which diminishes adsorption efficacy. Consequently, they often fail to meet the stringent requirements of industrial wastewater treatment systems. The limited specific surface area of clay particles poses challenges in their collection and regeneration from aqueous environments following adsorption. Furthermore, industrial wastewater treatment requirements are seldom met due to the poor affinity of hydrated clay for organic compounds, which leads to a significant reduction in adsorption efficiency upon reinforcement (Mukhopadhyay et al., 2020). Consequently, in order to address these indisputable facts, researchers globally have demonstrated considerable dynamism in the development of novel, sustainable bionanocomposite materials by integrating or imprinting significantly more active bio-based polymeric components (Rahman et al., 2023c). In this particular context, both synthetic and natural polymeric materials have been extensively employed. Among these, biobased natural polymers commonly referred to as naturally occurring polymeric materials have recently garnered significant attention due to their abundant availability and inherent biodegradability. These materials are increasingly recognized as pivotal in addressing contemporary challenges associated with "sustainable environment" initiatives and the principles of "green chemistry," particularly in their applications as adsorbents and biosorbents (Shahriar Kabir et al., 2018; Mukherjee et al., 2023; Rahman et al., 2018b).

Amongst all the remaining biopolymers, cellulose nanocrystals (CNCs) are sustainable nanomaterials that have great potential for treating industrial effluents or wastewater, offering a viable alternative to environmentally deleterious materials (Sadare et al., 2022; Rabby et al., 2025; Dewan et al., 2025). CNCs are needle-shaped or rod-like nanoparticles characterized by high crystallinity and dimensions typically below 100 nm which can be derived from various natural cellulose sources, including algal cellulose, bacterial cellulose, agricultural biomass, microcrystalline cellulose, tunicates, wood and cottons (Mali and Sherje, 2022). This novel class of biodegradable nanomaterial recently have garnered significant attention for their diverse applications due to their potential as a low-carbon footprint material (Tang et al., 2022; Rahman et al., 2024e). By 2025, the global nanocellulose market is projected to reach \$1.32 billion, driven by rising demand and innovative applications across industries. This growth reflects increased interest in sustainable materials and advancements in production technologies (Trache et al., 2020). Furthermore, the growing demand for nanocellulose and its innovative applications has spurred researchers and industries to capitalize on its potential, particularly in areas like water treatment, where its unique properties such as easy extraction methods, elastic modulus, large specific surface area, increased strength, biodegradability, crystallinity, good reaction activity, improved adsorption capability, and tunable surface chemistry offer sustainable solutions for environmental challenges (Rahman et al., 2024b; Jiao et al., 2024; Lu and Hsieh, 2010; Trache et al., 2017). Notably, cellulose nanocrystals (CNCs) exhibit exceptional adsorption capabilities against positively charged contaminants, including heavy metals, cationic dyes, and faintly positively charged antibiotics, as well as various organic and inorganic micro/nanopollutants (Mahfoudhi and Boufi, 2017, Batmaz et al., 2014; Shi et. at., 2022). This is because cellulose nanocrystals (CNCs) possess high surface area and negatively charged groups, such as carboxylates, hydroxyl groups which facilitate strong electrostatic interactions. These interactions, combined with hydrogen bonding and ion-dipole interactions, enhance adsorption efficiency by forming monolayer and multilayer structures depending on the density of the functional group (Lombardo and Thielemans, 2018). Meanwhile, modified natural clays can facilitate both monolayer and multilayer physisorption, as well as interparticular diffusion, owing to their high specific surface area and porous structure (Patel, 2019; Dey et al., 2016). This capability is attributed to the enhanced accessibility and adsorption sites provided by their structural characteristics. Over the past decade, there has been a burgeoning interest in developing clay CNC based nanocomposites that synergistically combine the beneficial properties of both components to optimize adsorption performance. The fabricated bionanocomposite exhibits a marked enhancement compared to its individual components modified clay powder or cellulose nanocrystals stemming from the synergistic combination of anionic hydroxyl (-OH) groups present in both clay and CNC, carboxylate groups inherent to CNC, and the mesoporous structure of the modified clay (Ayalew, 2022). The newly developed bionanocomposite exhibits exceptional hydrophobicity and adsorption capacity against a wide range of toxicants, including anionic, cationic, and non-ionic micro- and nanopollutants. These properties can be attributed to the synergistic effects that enhance its adsorption efficiency (Osman et al., 2023; Rahman et al., 2024a). However, for a better understanding of the scope, contribution, novelty, and potential of the newly proposed synergistic adsorption mechanism/exact pathway for the particular adsorptive separation of hazardous toxic pollutants from bulky wastewater bodies, Fig. 1 is provided. From the above figure, it has clearly been observed that the active binding sites that belong to the structure of the applied CNC-MC based bionanocomposites could be responsible for the formation of the particularly strong chemical bonding like coordination bond/covalent bond between the adsorbents binding sites and adsorbates via chelation mechanism by using the existing lone pair of electrons. Additionally, the porous structure of the fabricated bionanoadsorbents could be responsible for the formation of the monolayer and/or multi-layer physisorption, which was formed by some undeniable factors like hydrogen bonding, van der Waals's forces, dipole momentum, etc. Furthermore, the deep penetration or interparticular diffusion took place due to the presence of excessive pour tube and pour canal in the peripheral surface microstructure of the considered CNC-FC based biopolymeric nanoadsorbents during the selective separation of the hazardous toxicants like heavy metals and dyes from the bulky industrial wastewater bodies via fixed-bed column adsorption (Rahman et al., 2024a).

Despite numerous reviews on clay-polymer composites over the past few decades, the design of clay-CNC-based bionanocomposites utilizing their synergistic mechanism for real-time treatment of industrial effluents or wastewater remains unexplored. Developing these multifunctional bionanocomposites requires a thorough understanding of the efficient immobilization of CNC onto clay to leverage chemisorption, mono- and multilayer physisorption, and inter-particular diffusion mechanisms for large-scale applications. This review summarizes recent advancements in the development of natural clay-based bionanocomposites modified with cellulose nanocrystals and explores their potential applications in industrial processes, particularly for wastewater treatment. This review emphasizes on the cellulose nanocrystals extraction process, natural clay modification techniques, bionanocomposite fabrication and characterization, and their potential applications (by adhering to the synergistic mechanisms, including chemisorption, mono- and multilayer physisorption, and inter-particular diffusion). It also explores mathematical modeling and regeneration methods post-operation. A critical analysis of the performance of FC-CNC bionanocomposites is presented, alongside a comparison with commercial adsorbents. For greater clarity, future guidelines that are essential in this specific field of study have also been covered.

## 2. Main precursors, their sources, extraction, and modification

Treating industrial wastewater pollutants is the primary concern in environmental engineering, as well as developing a green biopolymer-based modern adsorption technique. That will be more effective in removing various industrial contaminants from water bodies. Because of their environmental harmlessness and degradability, polymer-based composites have gathered a lot of interest among the varieties of advanced adsorbents (Azimi et al., 2024; Bang et al., 2025; Castellanos et al., 2025; Norfarhana et al., 2024; Rahman et al., 2025a; Saqib et al., 2024). Some recent and most popular cited articles proved that nanocomposites can offer better chemical, physical, mechanical, morphological, microstructural, and compatibility characteristics over single polymers (Rahman et al., 2024d & 2025b). Where selecting materials like natural clay and CNC are the top choices among researchers because they possess uncompromising properties in terms of physicochemical and sustainability. They also have high surface area, and the potential for chemical modifications (Azimi et al., 2024; Bang et al., 2025, 2025; Das et al., 2022; Rahman et al., 2024c; Naseer et al., 2024; Rahman, 2024; Hossain et al., 2024; Sheikh et al., 2023a,b). Developing functionalized clay-CNC-based biopolymeric nanocomposites for wastewater treatment always relies on selecting appropriate precursor materials. Because lignocellulosic fibers and natural clays serve as the

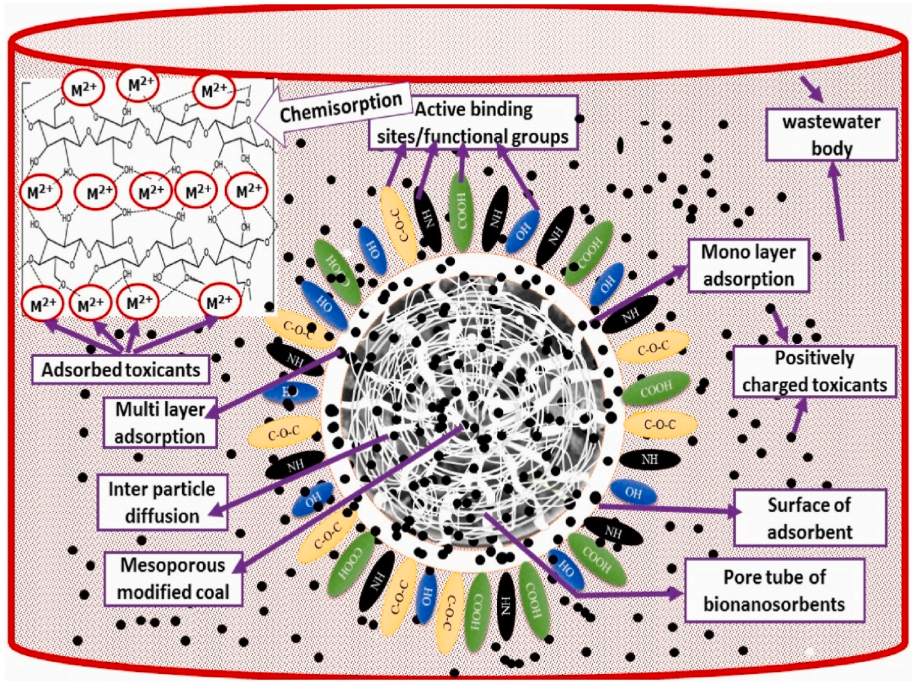



**Fig. 1.** Proposed synergistic adsorption mechanism including all the monolayer and multi-layer physical adsorption (due to hydrogen bonding, Van der Wall's forces, dipole momentum etc.), chemical adsorption due to coordination bond forming between the adsorbents binding sites and adsorbates via chilation), and inter-particular diffusion or deep penetration in the existing pour tube and pour canal of the considered CNC-MC based bionanocomposites (Rahman et al., 2024a). This content has been regenerated with the permission of Elsevier with license number 5998600199924.

fundamental building blocks, this section will discuss the sources, extraction methods, and modification techniques of lignocellulosic fibers and natural clays, emphasizing their role in enhancing adsorption efficiency and reusability for industrial wastewater treatment applications (Rahman and Maniruzzaman, 2021).

### 2.1. Lignocellulosic fiber sources and structures

Lignocellulosic fibers are promising for developing bionanocomposites due to their excellent abundance, renewability, and biodegradability. They have those characteristics because they are derived from natural plant biomass and consist of the most promising constituents, like cellulose, hemicellulose, and lignin, which are the main reasons for maintaining a distinctive combination of thermal constancy, mechanical strength, and compatibility with different polymer matrices. For better clarity, the distribution of the naturally occurring lignocellulosic fiber in the non-wood/softwood plants, including both primary and secondary plants, and their extraction, modification, chemical structure, as well as the production of CNCs and their potential application in various fields, have been shown **in** Fig. 2a and b. It can be said that cellulose, hemicellulose, and lignin actually provide the hierarchical structure of these fibers, which is key to their mechanical strength and functionality (Musa and Onwualu, 2024; Balasubramani et al., 2024; Stefanidis et al., 2014; Rabbani et al., 2024). Some other sources of lignocellulosic fibers are primarily derived from plant biomass, both primary and secondary (Fig. 2a), including agricultural residues, annual plants, and forest products. These abundant and renewable sources make them ideal for producing eco-friendly nanomaterials (Rahman et al., 2023c). The constitution of lignocellulosic biomass is a key factor in the formation of cellulose nanocrystals (CNCs). Therefore, understanding the formation of cellulose fibers inside lignocellulose is fundamental to optimizing CNC production. The selection of biomass is also important in terms of CNC production because biomass is rich in cellulose, and hemicellulose upholds higher CNC production. At the same time, a higher lignin content can decrease CNC yields (Qureshi et al., 2024). So, biomass sources with low lignin and high cellulose content typically generate the most considerable amounts of cellulose nanocrystals (CNCs). Cellulose is the most abundant component in lignocellulosic materials, providing strength and rigidity to the fibers. It can be found in the form of microfibrils, which significantly influence the mechanical properties of these fibers (Z. Zhang et al., 2024; Fernando et al., 2023; Fang and Catchmark, 2014; Manian et al., 2021). Hemicellulose acts as matrix that binds cellulose fibers, contributing to the biomass's flexibility and water retention properties (Albornoz-Palma et al., 2025; Falade et al., 2025; Zoghlami and Paës, 2019). Where lignin provides structural support and protection against microbial attack. However, its presence can hold back the processing of lignocellulosic fibers due to its resistance (Sun, 2020). For better understanding the overall arrangement and chemical structure of the lignin, cellulose, and hemicellulose in the naturally occurring raw lignocellulosic fibers have been shown in Fig. 3 and Table 2.

#### 2.1.1. Extraction of raw fiber

The extraction of raw fibre involves various methods and techniques, which are designed according to the types of plants and properties of the extracted fibres. The extraction process generally includes mechanical, chemical, or a combination of methods to separate fibres from plant matrices. They are often used to enhance the properties of fiber for a specific application. This process significantly enhances the fibers' quality, yield, and characteristics, which are essential for their application in textiles, composites, and other industries (Rahman et al., 2023b). Moreover, recently developed extraction methods have been known for being efficient and environmentally friendly, enhancing fiber yield and preserving the fibers' integrity. Such as enzymatic treatments, bacterial decompositions, water retting and due retting are gaining traction for improving the quality of extracted fibres while minimizing ecological impact (Rahman and Maniruzzaman, 2021; Hossen et al., 2024). The method that needs to be chosen to extract fiber often relies on the anticipated properties of the extracted fiber, which can be its thermal stability, mechanical strength, and chemical composition

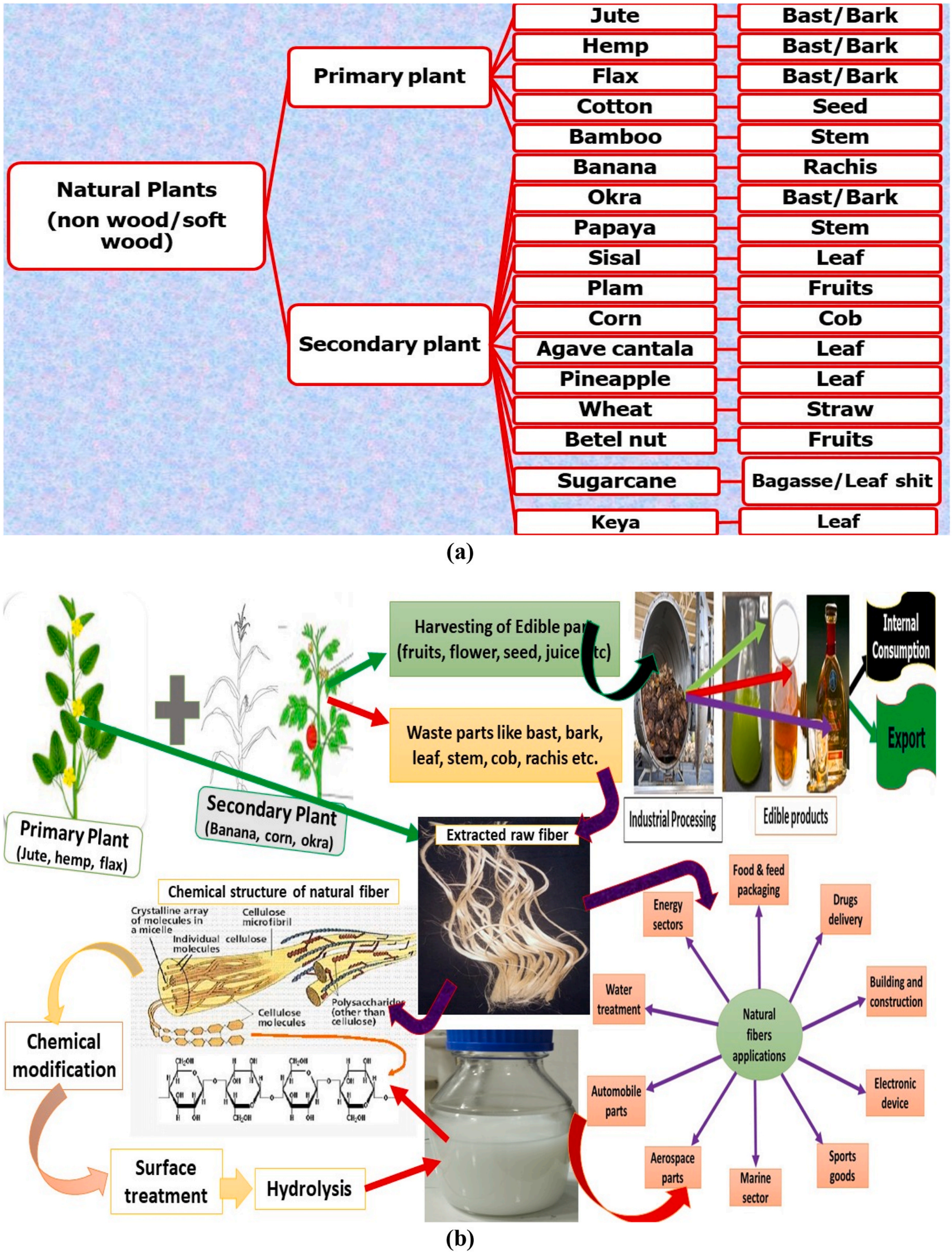



**Fig. 2.** (a) Distribution of naturally occurring raw lignocellulosic fiber in the non-wood/softwood plants including both primary and secondary plants, and (b) Extraction, modification, chemical structure, as well as production of CNCs and their potential application in various fields.

(Rahman et al., 2024g; Hossain et al., 2024; Sheikh et al., 2023a,b; Rabby et al., 2025; Dewan et al., 2025). These studies discussed that eco-friendly retting techniques can effectively extract raw lignocellulosic fibers from various primary and secondary plants sources such as banana rachis fibers, agave cantala leaf fibers, keya leaf fibers, sugarcane leaf shit, maize shell, corn cob, etc. To do this, a banana stalk, about 10–12 inches in size, is submerged underwater for 4 (four) weeks or around 30 days. This process allows for complete bacterial decomposition and helps isolate the raw banana rachis fibers. During this period, the bast material decomposes fully, loosening the fibers. Afterward, the rachis fibers are washed multiple times with fresh water to obtain the best-quality raw lignocellulosic fiber. The fibers are then dried either in

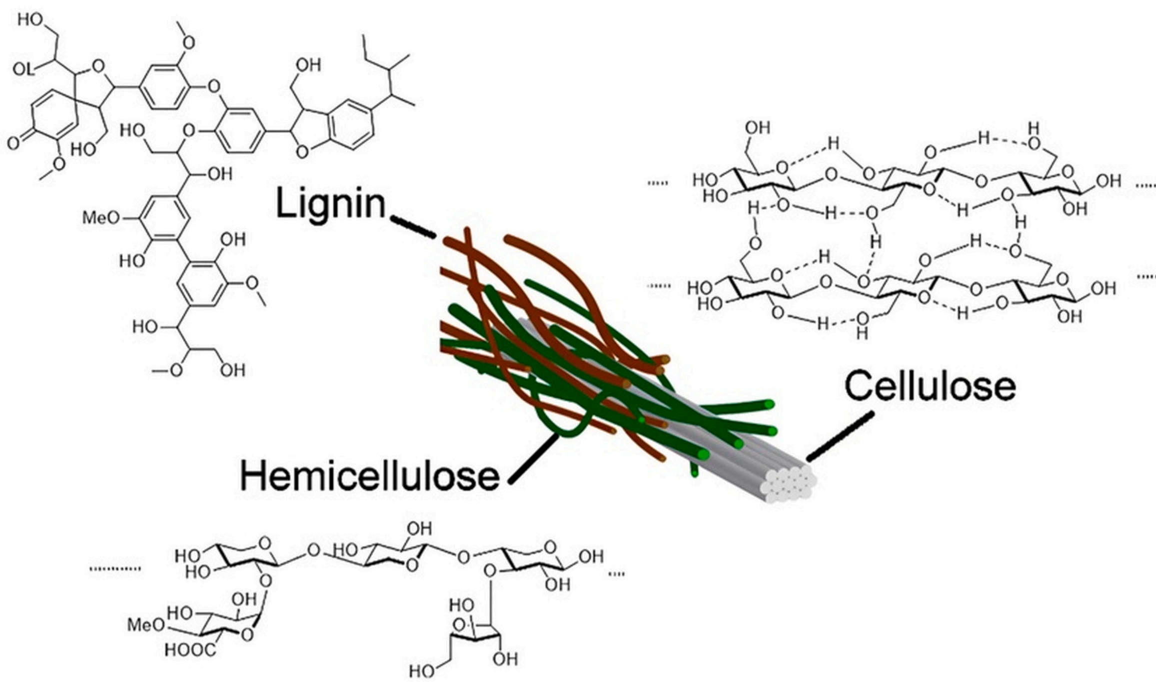



**Fig. 3.** The complex structure of lignocellulosic fibers (adopted from T. Li and Takkellapati, 2018).

sunlight or in a dryer until completely drying as well as they are ready for further processing. The process may include processes like scouring, alkali treatment, and acid hydrolysis for the production of CNC. Also, Rahman and co-workers have extracted Agave cantal leaf fiber with the same process by conducting the prominent water retting method. Whereas after 9–10 days of immersion, the leaves significantly degraded, allowing for the extraction of the fibers (Rahman et al., 2023b). Rabby and co-workers have extracted raw maize(shell/cob) fiber by conducting mechanical hammer milling and blending methods because of its several advantages, i.e., it is fast, easy, cost-effective, and compact. During the milling process, the size of the shell is reduced as much as possible to facilitate further processing. After the milling process, the raw fiber is dried in an electric vacuum oven manufactured by a German company named BINDER GmbH at about 105 °C for about 2 h. Once dried, the raw fiber is collected for further use (Rabby et al., 2025; Hassan et al., 2024) Discuss how the fibers from the leaf sheaths of mature sugarcane (Saccharum officinarum) can be extracted. First, the plants and leaves and their sheaths are cut down separately to facilitate the mechanical blending process. Next, the leaf sheaths are cut into segments measuring 5–6 cm using scissors and then blended in a Jaipan blender to improve fiber accessibility.

### *2.1.2. Modification of raw lignocellulosic fibers*

Raw lignocellulosic fiber modification is a crucial process to enhance the properties of fiber for various applications. Fiber goes through a process that alters the chemical structure to improve its compatibility with other materials, increase its mechanical strength, and enhance its thermal stability. Recently, various methods have been introduced by researchers, including chemical treatments and advanced pretreatment techniques that have been explored to achieve these modifications. Below are some key approaches and findings from recent research on lignocellulosic fiber modification. Alkali treatment, by using sodium hydroxide, is a common practice to modify lignocellulosic fibers. This process helps to remove impurities and increase cellulose content. Also, there is evidence of enhancing mechanical strength and thermal stability (H. Chen et al., 2021; Kumar et al., 2024; S. Liu et al., 2024; M et al., 2024; Zhong et al., 2021). For instance (M et al., 2024), study shows how alkali-modified Sunn hemp fibres improved properties and antimicrobial activity against bacteria like Staphylococcus aureus. L. Zhang et al. (2024) study plays an alkali treatment with γ-Aminopropyl triethoxysilane. This dual modification helps to enhance interfacial compatibility with polymers, and in composites with better flexibility, tensile strength, and water resistance. Organosolv and Enzymatic Pretreatments is a technique that is often combined with lignin modification strategies and is much effective in fractionating lignocellulosic biomass. For biofuel production, this method helps to reduce lignin content, by facilitating enzymatic hydrolysis and increasing sugar yield. For example, acidic butanediol pretreatment with lignin repolymerization inhibitors significantly improved glucan conversion to fermentable sugars (Xie et al., 2024). Song et al. (2024) perform an in situ lignin modification through organosolv pretreatment. where polyethylene glycol is used, which helps to enhance enzymatic hydrolysis by changing lignin's physicochemical properties. Some advanced chemical modifications are also in practice, such as Iodization and other chemical modifications, which can enhance the properties of lignin-based materials (Han et al., 2023).The paper discusses the iodization modification strategy for lignin-based carbon fibers, enhancing their surface area and graphitization degree. This method increases the π electron cloud density in the lignin structure, improving π-π interactions (supramolecular bonds) between lignin molecules. Which also improves thermal stability and graphitization, which is beneficial for applications like supercapacitors (Spiliopoulos et al., 2024). modify cellulose properties by exchanging functional cations, such as lanthanides, cellulose nanocrystals can acquire new functionalities, including optical and magnetic properties.

### *2.1.3. Production of CNCs from the previously modified natural lignocellulosic fibers*

Cellulose nanocrystals (CNCs) are biodegradable, renewable, and versatile nanomaterials derived from cellulose, which is the most abundant biopolymer on this globe/planet. Their intensive properties make them suitable for various applications, including biocomposites, food packaging, and medical devices. In the most naturally occurring cellulosic materials, the internal structure of the cell wall of each fiber bundle predominantly consists of cellulose, hemicellulose, and lignin. To isolate pure cellulose and convert it to CNCs, it is essential to properly

**Table 2**
The lignocellulosic contents, sources, potential applications, and chemical composition of different types of natural fiber (based on their source and high cellulose value) are abundant in Bangladesh.

| Fiber Type | Source | Structural properties (celluolose/ lignin) | Potential Applications | References |
|---|---|---|---|---|
| Banana Rachis | Banana pseudostem | High cellulose (~63 %) low lignin (~8 %) | Biodegradable composites, textiles, paper, waste water treatment | (Jatkar et al., 2024; Provin et al., 2024; Rabby et al., 2025; Rahman et al., 2022, 2024f; Sheikh et al., 2023a,b) |
| Hemp Fiber | Hemp stalks | High cellulose (~76 %), and lignin ((~12 %), strong fibers | Bio-composites, reinforcement in plastics, automotive industry | (Pepi et al., 2024; Rabby et al., 2025; Rahman et al., 2023b) |
| Flax Fiber | Flax plant stems | cellulose (~75 %), and lignin((~4.8 %), High tensile strength, long fiber length | Eco-friendly textiles, bio-composites, automotive industry | (Amiri et al., 2017; Calabrese et al., 2025; Perera et al., 2025; Rabby et al., 2025) |
| Jute Fiber | Jute plant stems | cellulose (~61 %), and lignin((~13 %), good flexibility | Ropes, sacks, geotextiles, biocomposite, biopolymer, automotive industry | (Arunachalam et al., 2025; Rabby et al., 2025; Shahinur et al., 2022; Taiwo et al., 2025) |
| Coir Fiber | Coconut husk | cellulose (~43 %), and lignin((~45 %), High lignin, rigid structure | Mattresses, ropes, brushes,bio brick, cementing composite | (Ajiz et al., 2024; Martinelli et al., 2024; Rahman et al., 2023b; Thakur et al., 2021; Zheng et al., 2025) |
| Bamboo Fiber | Bamboo culms | cellulose (~73 %), and lignin((~10 %), High cellulose content, lightweight, durable | Bio-composites, furniture, textiles, Paper industry | (H. Li et al., 2024; Mohammed et al., 2024; Rabby et al., 2025; Rahman et al., 2023b) |
| Sugarcane Bagasse | Sugarcane processing waste | High cellulose (~40–50 %), moderate hemicellulose | Packaging materials, biofuel production | (Charoensopa et al., 2024; Mubarak et al., 2024) |
| Sugarcane leaf sheath | leaf sheath | cellulose (~54 %), and lignin((~11 %), | CNC (Cellulose Nanocrystal), biocomposite | Hassan et al. (2024) |
| Wheat Straw | Agricultural residue | cellulose (~45 %),High hemicellulose (~35 %)), medium lignin(~26 %), | Animal bedding, pulp and paper, bio-composites, food packaging | (Bangar et al., 2023; Lamm et al., 2024; Serrano et al., 2024; Zhang et al., 2022) |
| Cotton Fiber | Cotton plant | Nearly pure high cellulose (~95 %), low lignin (~1 %) | Textile industry, high-strength bio-composites | (Naoumkina et al., 2024; Sharif et al., 2024; Shi et al., 2024; Yang et al., 2024) |
| Agave Fiber | Agave plant leaves | High cellulose (~70 %), low lignin (~10 %) | Bio-based polymers, textile reinforcement | (Lalaymia et al., 2025; Lazaro-Romero et al., 2024; Rahman et al., 2023b; Sumarago et al., 2024a) |
| Okra Fiber | Okra plant stems | cellulose (~70 %), lignin (~15 %) | Bio-composites, food packaging, biodegradable films | (Lv et al., 2024; Rahman et al., 2024b; Zhang et al., 2024) |
| Rice Husk | Rice milling byproduct | High silica, moderate cellulose (~35–40 %),lignin (~25 %) | Silica-based materials, paper industry, acid hydrolysis treatment, wastewater tretment | (Hafid et al., 2021; Rahman et al., 2025a) |
| Maize Cob | Corn processing waste | High cellulose (~45–55 %), lignin (~15 %) | Bio-based polymers, bio composite | (Dewan et al., 2025; Rabby et al., 2025) |
| Keya (*Pandanus tectorius*) | Pandanus tree leaves | High fiber strength, good water resistance cellulose (~54 %), lignin (~12 %) | crystalline nanocellulose,bio nanocomposite | Hossain et al. (2024) |

clean, eliminate, and remove lignin and other impurities. Because these impurities often introduce an amorphous nature to cellulosic biomass. To achieve this, defined chemical processes are employed stepwise, with scouring, alkali treatment, bleaching, and acid hydrolysis (including both single and double). Here, scouring is removing dirt, oil, dust, and gummy or waxy matter from raw fibers using a soap solution. The specific parameters for this process, such as the soap-to-fiber ratio, temperature, and final steps, may vary depending on the type of fiber and its intended use. However, the main techniques are the same for all. For example, Rabby and co-workers have shown how scouring on maize (*Zea Mays*) shell fiber is carried out to produce CNCs. In the process, they used a ratio of the considered fiber to liquid soap solution around 1:15, raw fiber was scoured with a 2 % soap solution by gently stirring it at room temperature for about 1 h. After performing a quality scouring process, the fiber was washed properly with distilled water, and then the fiber was dried using an electric vacuum oven. Whereas the drying temperature was maintained at 105 °C for 2 h. To produce cellulose nanocrystals (CNCs), the process involved limiting the fiber's connection with the alkali solution and applying the following chemical treatments (Rahman et al., 2024b) used untreated okra stalk fibre and treatment with a 5 % soap solution. The temperature is maintained at 60°C for 2 h, to scouring of raw rachis fiber. The cellulose sample was mixed with a soap solution at a ratio of 1:10 in a 2-liter borosilicate glass beaker. The ratio of soap solutions is 7.5 % (w/v). In the process, the temperature was maintained at 65 ± 0.8 °C with gentle agitation, and the process was performed for 2 h (Dewan et al., 2025). conducted a scouring process to remove different dusty, waxy, gummy, and oily substances from the maize cob fiber, which was carried out using a 5 % soap solution. The ratio of the soap solution was 5 % with a ratio of 1:20 at a temperature of 60 °C. The scouring was operated for 2 h with moderate stirring by maintaining the temperature. After the scouring process, alkaline treatment is the standard practice for researchers. This method involves using alkaline solutions, such as sodium hydroxide or potassium hydroxide (Hassan et al., 2024). The aid of the process is to eliminate the maximum amount of alkali-soluble substances from the fiber and to reveal the short length of the crystallites more clearly (Rahman et al., 2024d). The process is commonly carried out to enhance the crystallinity and mechanical properties of fiber (Devi et al., 2022; Rahman et al., 2024e). The alkaline treatment process is designed for a special task here to improve the breakdown and dissolution of lignin matter. Again, it also facilitates the hydrolysis of hemicellulosic complex structures (Dewan et al., 2025). The process clarifies the cellulose structure and also helps to enhance its accessibility for subsequent treatment. Because of the treatment carried it disrupts the bonds between oxygen and hydrogen(OH) in the fiber structure, which turns the ionization process of hydroxyl groups to produce alkoxide (Ng et al., 2015; Hassan et al., 2024).

$$\mathrm{NaOH} + \mathrm{Fiber} - \mathrm{OH} \rightarrow \mathrm{Fiber} - \mathrm{O}- \mathrm{Na}+(\mathrm{Alkoxide}) + \mathrm{H2O} \quad (1)$$

(Dewan et al., 2025; Rabby et al., 2025) Conducted the treatment using a 16 %–18 % sodium hydroxide (NaOH) solution. Where the volume ratio was 1:15/1:18 of fiber, the mixture was stirred moderately on a hot plate at temperatures ranging from 50 to 80 °C for a duration of 1–4 h. It usually depends on the type of fiber. After the treatment, the residue was dried using an electric vacuum oven. The temperature of the oven was 105 °C and lasted for 2 h. By conducting this procedure, they extract maize cob fiber and maize (*Zea Mays*) shell fiber. Some Well-known strong bases like aqueous sodium hydroxide, ammonium hydroxide, calcium hydroxide, and potassium hydroxide are commonly used for this technique. (Thandavamoorthy et al., 2023). Among them, sodium hydroxide is commonly used because it is popular for its effectiveness. However, there are some environmental concerns. It produces environmental hazards (Raharjo et al., 2024; Bichang'a et al., 2024; Malakar et al., 2025; Thandavamoorthy et al., 2023; Mbisana et al., 2024). A bleaching process of alkali-treated fiber was performed to achieve complete lignin removal. The fiber is immersed in a mixture of multiple chemical solutions during the process. Where pH is an important factor here. It is essential to maintain the pH level of a basic or acid solution at 4 during this treatment. It is essential to protect the fiber's functional properties. For the fiber treatment, a 2 % sodium chlorite ($NaClO_2$) solution and a 2 % sodium metabisulfite ($Na_2S_2O_5$) solution were used, and to achieve a stable pH in the solution, either 0.1 N hydrochloric acid (HCl) or 0.1 N sodium hydroxide (NaOH) was used (Dewan et al., 2025). The temperature was maintained properly at 95 °C and lasted 2h with a moderate stirring. According to (Hassan et al., 2024), in acidic conditions, $NaClO_2$ releases chlorine dioxide ($ClO_2^-$) as an alternative to chlorine. This bleaching solution efficiency relies on the rate at which $ClO_2^-$ is generated, as it can oxidize lignin residues left in the fibers by targeting their aromatic ring structures. Following this, the alkali-treated (dried) raw fiber was treated with a 2 % $Na_2S_2O_5$ solution, where the fiber-to-solution ratio was 1:30 and with continuous stirring. After treatment, they were repeatedly washed with distilled water to neutralize any residual bleaching compounds on the fiber. The procedure was carried out in a dark environment for approximately 30 min (Hossain et al., 2024; Rahman et al., 2024g; N et al., 2024; Sheikh et al., 2023a,b). The study found that the solution treated with $NaClO_2$ initially displayed a yellowish color. Afterward, the fiber was correctly rinsed multiple times with distilled water and became mostly white. It was then dried in an electric oven, where the temperature was maintained at 105 °C. Bleached alpha-cellulose must be cut into small pieces (as possible) for the acid hydrolysis process. Following this, the CNCs were produced through the acid hydrolysis of the bleached small-sized fibers (Dewan et al., 2025; Sartika et al., 2023). perform the hydrolysis process by using 60 % $H_2SO_4$ solution with nonstop stirring on a hot plate. The temperature was maintained here at 40 °C for a duration of 15 min with a continuous magnetic stirrer. Where the fiber and solution were maintained at a 1:15 ratio in a reaction beaker which placed on a magnetic hot plate (Brand: LABCOM, Model-MS-1003) with continuous stirring. The procedure was performed by stirring at the highest speed (around 1500 rpm with a tolerance of ±5 rpm) for about 20 min until a deep brown color was observed and all visible particles were eliminated. To maintain the proper properties of the newly produced CNCs, this study employed ice blocks for a quick quenching of the hydrolysis reaction. A sophisticated centrifugation tool manufactured by Germany (Model: MIKRO 22R, Hettich ZENTRIFUGEN) was utilized for a duration of 15 min at the highest speed of 6000 ± 15 rpm, within a temperature range for each batch of 1–5 °C. The quenching step is crucial to this process; if quenching occurs before the formation of nanostructures, the considered cellulose/cellulosic fibers would not be reached the nanoscale (1–100 nm). Conversely, cellulose may ignite and be destroyed if the quenching process is delayed. The outcome produces either carbon dots exclusively or a combination of carbon dots and cellulose nanocrystals (CNCs) (Rabby et al., 2025). Finally, after being fully neutralized to a pH of 7.0 using distilled water, the solid fraction of CNCs was stored in 96 % ethanol (Sheikh et al., 2023a,b; Kusmono et al., 2020). However, this process also generates significant acidic wastewater, posing environmental concerns (Balasubramani et al., 2024; Binczarski et al., 2024; Lv et al., 2024; N et al., 2024; Sumarago et al., 2024). To overcome this problem the prominent Deep Eutectic Solvents (DES) methods are introduced by researchers (Firouzi et al., 2025; Baraka et al., 2024; Huang et al., 2024). It's a more eco-friendly alternative; DES combined with ultrasound assistance has been used to extract nanocellulose effectively by removing lignin and hemicellulose while reducing chemical usage (Kaur et al., 2024; Sharma et al., 2025; Meraj et al., 2024). A study about the extraction of the nanocellulose from durian husk fibers using DES-based extraction with in situ ultrasound assistance achieved a high yield of 58.22 % and effectively removed lignin and hemicellulose (Lim et al., 2024). However, to enhance clarity, Schematic flow diagram of the extraction of CNCs from the naturally occurring plant waste biomass via applying various chemical and biochemical processes (general), and Photographical representation of the production of CNCs from the waste biomass of secondary plant namely banana tree rachis derived raw lignocellulosic fiber during the experimental session as per Rahman et al. (2024d) have been shown in Fig. 4a and b.

### 2.2. Natural clay and its modification techniques

Natural clay is a group of fine-grained earth materials primarily composed of hydrous aluminum silicates. It has some special properties, which make it a valuable material for diverse applications. They have a high surface area, cation exchange capacity, and layered structure (Yang et al., 2024; Hu et al., 2024; Xie et al., 2022). Its modification enhances its properties, making it more effective for specific uses (Rahman, 2024). Various techniques have been developed to modify natural clay, each with unique benefits and applications (shown in Table 3) (Rahman et al., 2024f; Azimi et al., 2024; Hussain et al., 2024; Naseer et al., 2024). These modifications aim to improve adsorption capacity, chemical stability, and structural properties, thereby expanding the utility of clay in different fields. Some popular modifications that were done by the researcher will be discussed in this section. There are several types of natural clay, each with some special properties that make them popular in a wide range of applications. Montmorillonite is Known for its high cation exchange capacity and expandable structure, it is widely used in adsorption applications (Liu et al., 2024; Sarkar et al., 2024). Another versatile clay used in environmental remediation and industrial processes due to its adsorption and catalytic properties is bentonite (Chen et al., 2024; Zhang et al., 2024). Kaolinite is common in ceramics and as a filler in paper and plastics, it has a less expandable structure compared to montmorillonite (Gounden et al., 2024; Mazitova et al., 2022; Thue et al., 2023) This paper focuses on the modification of natural montmorillonite clay using $H_2O_2$ to enhance its adsorption capacity for Acid Red 114 dye. Here, natural clay undergoes a chemical treatment by oxidation with H2O2, which increases oxygen content and introduces diverse surface functional groups. Thus, it improves the clay's hydrophilicity and porous structure, leading to significantly higher adsorption performance than untreated clay. The study emphasizes how this modification can be vital in the application of water treatment purposes (Kussainova et al., 2024). research on how to modify natural bentonite clay using polyhydroxocations of iron (III) and aluminum (III) through the "co-precipitation" method. This modification alters the clay's chemical composition, structure, and adsorption properties, this process creates porous, nanostructured materials that have a greater ability to absorb bichromate and arsenate anions, which improves its functionality for environmental applications. (Rahman, 2024) discuss in his paper how chemically modified clay brings benefits in terms of water purification applications, such as acid activation, organic-inorganic solvent treatment, organoclay formation, and the cation exchange method (Hamdi et al., 2024). investigates three natural clay samples, AM (51 % calcite), HJ1 (32 % kaolinite), and HJ2 (32 % microcline). Modification techniques are implied through the use of acid-activated and base-activated clays, which were tested alongside natural clays. The main aim of this modification is to enhance the adsorption capacity of clays for removing salinomycin from environmental compartments. They conducted an experiment in batch form with 0.5 g of clay adsorbent and mixed with increasing doses of salinomycin (SAL) solutions of about 10 mL. The contract duration was 24 h, and the temperature was maintained at room temperature. The adsorption/desorption of salinomycin was characterized using an HPLC-UV instrument. The process was carried out across three separate pH ranges. For acid-activated clays, the

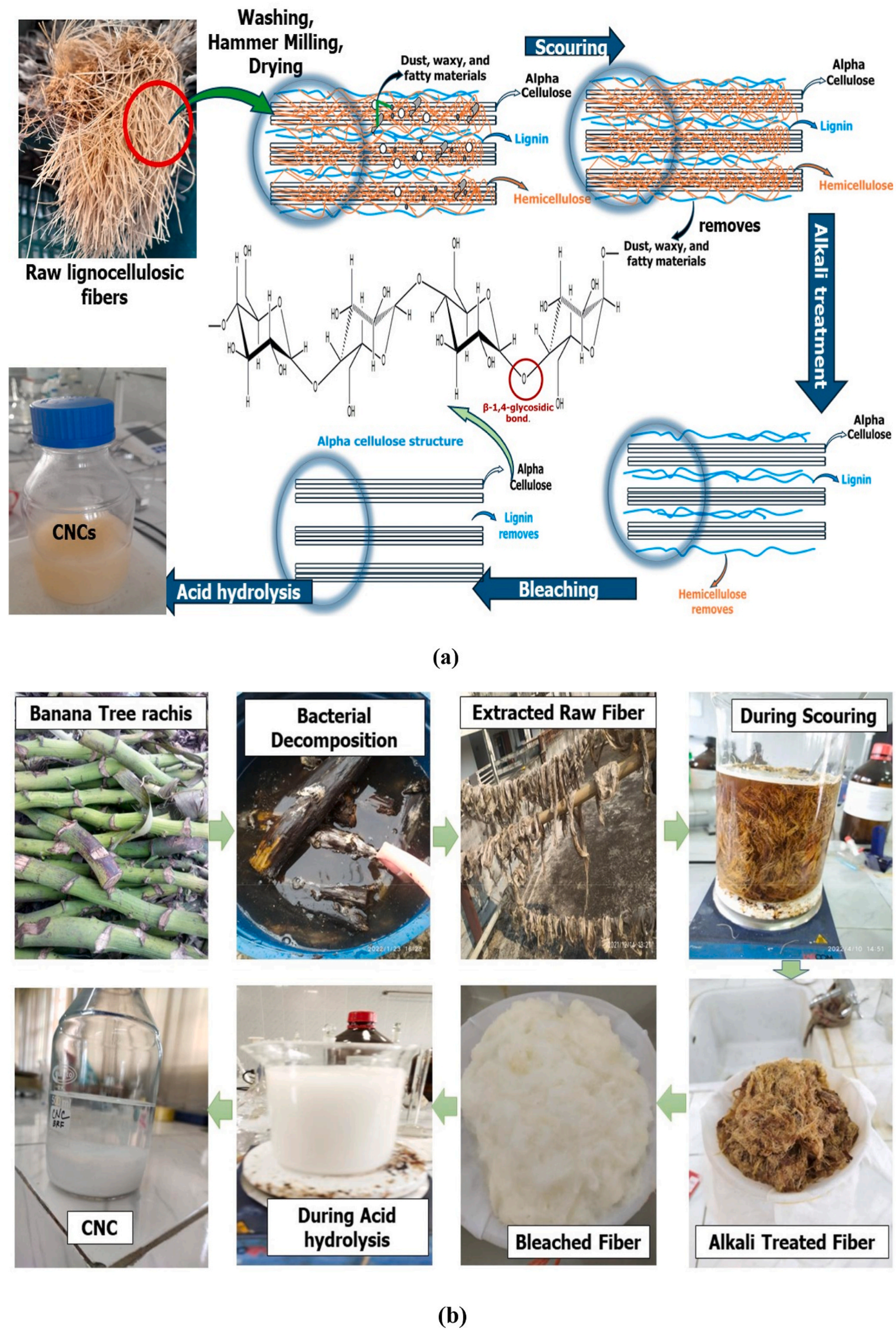



**Fig. 4.** (a) Schematic flow diagram of the extraction of CNCs from the naturally occurring plant waste biomass via applying various chemical and biochemical processes (general), and **(b)** Photographical representation of the production of CNCs from the waste biomass of secondary plant namely banana tree rachis derived raw lignocellulosic fiber during the experimental session as per Rahman et al. (2024d). This content has been regenerated with the permission of Elsevier with license number 5998590621982.

range is 3.33–4.49; for natural clays, it is 8.39–9.08; and for base-activated clays, the range is 9.99–10.18.

## 3. Fabrication process of CNC-clay nanocomposites

CNC-clay nanocomposites are used to create a hybrid material where CNC and clay are attached together to get the desired barrier properties as well as thermal and mechanical behavior. In case of environmental remediation like waste water treatment, has been extensively studied. There are different categories of nanocomposites but here, the popular fabrication process of polymer-based bio nanocomposites has been summarized.

### 3.1. Solution/solvent casting method

The solution/solvent casting method is a straightforward and versatile technique. Based on the desired properties of the final product appropriate polymer, fillers (clay) and solvent have to be chosen initially. Selected Polymer dissolved into compatible solvent. Achieving a homogenous mixture often requires heating or stirring (Rahman et al., 2025b). Ultrasonic treatment or mechanical stirring to achieve a uniform dispersion. There is a relation between specific surface area and

**Table 3**
Applications and Modifications of Natural Clay along with the key outcomes.

| Application Area | Modification Technique | Key Outcome/results | References |
|---|---|---|---|
| Heavy Metal Removal | Acid modification ($H2SO4$, HCl, KH2PO4) | Enhanced adsorption capacity for Fe(III), Zn(II), chromium (VI), Cd(II), Pb(II), Hg(II) | (Dim et al., 2021; Emam et al., 2016; Ndé et al., 2019) |
| Dye Removal | Surfactant modification | High removal efficiency (>90 %) for cationic dyes | (Bagheri et al., 2024; Bagheri et al., 2024; Soni et al., 2018) |
| Radionuclide Sorption | Organic functionalization | Improved removal of radioactive contaminants | (Jiménez-Reyes et al., 2021; Kyzioł-Komosińska et al., 2025; Maulden et al., 2024; Singh and Um, 2023; Yamaguchi et al., 2024) |
| Lithium Battery Anodes | Natural clay-based materials | Stabilized interface, dendrite-free operation | (Lan et al., 2021; Wang et al., 2025) |
| Catalytic Applications | Pillaring and intercalation | Enhanced thermal stability and catalytic activity | Barakan and Aghazadeh (2021) |

adsorption rate. As the surface area increases, the adsorption rate also increases (Adeyemo et al., 2017). For this purpose to get more adsorption rate clay minerals has to go through different physiological and chemical operations. For the effective fabrication of functionalized clay CNC-based nanocomposite, combine the clay-CNC suspension with the selected polymer solution. High-shear mixing or sonication to ensure a uniform distribution of the CNC and clay within the polymer matrix. The produced polymer solution has generally been poured onto a casting tray or Petri dish spreading it evenly. Solvent that is within the polymer solution has to evaporate keeping it in ambient temperature and subsequent pressure. For better efficiency of removal of solvent it could be placed in an electric vacuum oven to conduct the prominent technique namely solvent evaporation induced phase separation (EIPS) operation. Rahman and coworkers have done their experiment for the effective fabrication of the CNC-AC based multifunctional superactive biopolymeric nanosorbents by applying this prominent EIPS technique to get better yield and greater efficiency. While they used a special type of electric vacuum oven and kept the freshly prepared CNC-AC biopolymeric complex mixture in a liquid/semi-liquid phase for a long time around 12 h at a fixed temperature of around 60°C (Rahman et al., 2024d). One of the disadvantages is that the solvent evaporation step can be lengthy, particularly for thicker films. The next step is when the freshly prepared bio-nanocomposite is dried then it is peeled off from the Petri dish/tray. For increasing additional property, additional treatment may be required. Newly fabricated film is further crushed to produce fine nanoparticle then it is used for different beneficial applications (Qi et al., 2017). However, for a better understanding, a schematic flow diagram addressing the solution/solvent casting method has been shown in Fig. 5a and b.

### 3.2. In-situ polymerization

One of the promising methods of fabrication of biopolymetric nanocomposite is the in situ polymerization technique that creates advanced materials by the combination of different properties of organic and inorganic components. This process involves the dispersing of polymer and nanofiller in a suitable solvent so that stable suspension creates where uniform distribution occurs (Rahman, 2024). In the suspension, later on initiators are introduced, which increases the rate of polymerization reaction while controlling the reaction conditions such as heating or exposure of UV light. The in situ method retains uniform composite structure while enhancing the interfacial interaction between reactants and, in the meantime, doesn't allow the agglomeration of inorganic particles in polymer matrices (Yang et al., 1998). When polymerization is completed, then the final bio nanocomposite is subjected to post-processing procedures where washing, drying, and subsequent removal of excess solvent is mandatory. The final product that is produced because of their enhanced barrier properties, thermal stability and mechanical strength it is suitable for many applications like wastewater treatment, biomedical applications, and biodegradable materials. However, for a better understanding, a sketch diagram regarding the in situ polymerization technique has been shown in Fig. 6.

### 3.3. Spin coating method

Another popular fabrication technique is spin coating to produce a biopolymeric nanocomposite, which provides a controlled and uniform film-like nanocomposite. Firstly, a suitable volatile solvent has to be chosen where polymer and nanofiller are often combined with a polymer matrix (Rahman et al., 2025b). Dispersion of the components is crucial for achieving the desired mechanical and thermal behavior. After the dispersion, the sample is placed on a clean substrate basically a glass plate. A spin coating machine is used namely a spin coater where substrate is placed on, and it's rotated with a predetermined velocity. Rotation continued until the desired film thickness is achieved as well as solvent evaporates simultaneously at that moment (Fortunati et al., 2014). This process can be enhanced by crosslinking and strengthening the film. The crucial factor for improving the thickness of the final products is to maintain the concentration of the solvent and solution (Tyona, 2013). The resulting bionanocomposite film exhibits improved mechanical properties due to the reinforcement provided by the polymer and nanofiller. Spin coating is a useful method for creating sophisticated bionanocomposite materials because it gives exact control over the thickness and content of the film. It is important to mention that the whole process has been shown in Fig. 7 with a sketch flow diagram.

### 3.4. Electrospinning method

Electrospinning is one of the well-established fabrication methods of advanced biopolymeric nanocomposite with notable applications in biomedical, biodegradable polymers, drug delivery, and tissue engineering. Before starting the process, electrospinning solution preparation is a crucial step in dispersing clay and CNC in a suitable solvent. For uniform dispersion, ultrasonication and magnetic stirring are applied. Sometimes, to enhance the properties of nanocomposite polymers, it is added. The main parts of the electrospinning system are, firstly, a high-voltage source that will generate an electric field, then a syringe pump, and finally a rotary collector disk (Ashori et al., 2019). After mixing modified clay, CNCs, and other polymers/additives, the electrospinning solution is placed into the syringe, which is attached to a metallic needle. Between the grounded collector and needle, a relatively high voltage is applied; as a result, the produced electric field could turn the solution into fine jets. The nanocomposite is deposited on the collector while the solvent evaporates. Voltage, solution viscosity, and distance between needle and collector are crucial for the desired properties of nanocomposites. Newly produced nanoparticles will adhere to the polymer solution that is influenced by the solubility of the existing polymer in solvents, dispersion of CNC in solvents, and adsorption of polymer (Gong et al., 2022). However, for a better understanding, a schematic flow diagram addressing the electro spinning method has been shown in Fig. 5b.

### 3.5. Dip coating method

One of the versatile and widely used low-cost fabrication techniques

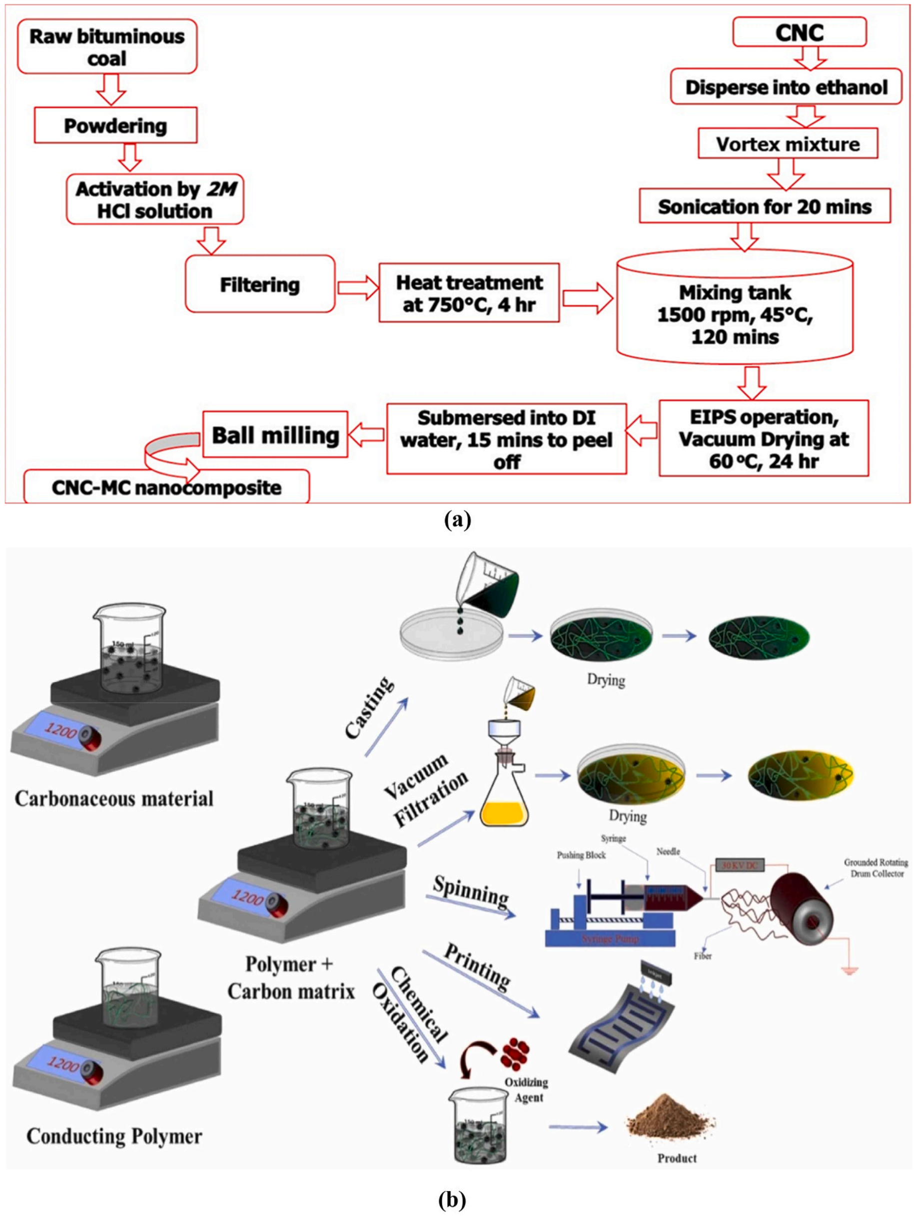



**Fig. 5.** Procedure for fabrication of bionanocomposite by means of (a) solution casting and EIPS techniques, and (b) spin coating, electro spinning, 3D printing, solvent blending method (Rahman et al., 2024a & 2025b) and this content regenerated with the permission from Elsevier with a license number 5998600199924 and 5999400938612.

of polymer/biopolymer-based nanocomposite film is the dip coating method. Making a nanocomposite solution, which consists of dispersing nanoparticles (such as metals, ceramics, or polymers) in an appropriate solvent, is the first step in the process. Careful formulation of the solution is required to ensure proper dispersion of the nano biopolymer. The substrate for the solution needs to be cleaned thoroughly so that no impurities affect it. During the fabrication process, the substrate is first immersed in the solution and then withdrawn at a controlled speed. A slower withdrawal produces thicker films, and higher speed produces thinner films, though thickness depends on different factors like the viscosity of the solution and concentration of nano biopolymer, temperature, and humidity (Nguyen-Tri et al., 2018). After the substrate is withdrawn from the solution, drying is ensured to allow the solvent to evaporate, and as a result, a solid nanocomposite film is produced (Dey et al., 2016). Noteworthy that nanocomposite films produced by this method exhibit increased mechanical strength and good chemical stability. Because of its outstanding properties, it is widely used in various fields like wastewater treatment as an adsorbent for the selective separation of the toxic pollutants via fixed bed column continuous adsorption process for better findings.

### 3.6. Sol-gel method

The sol-gel method is one of the extensively used fabrication techniques for biopolymeric nanocomposites like CNC-FC/Chitosan-AC based bionanoadsorbents, where a bioderived polymer matrix is reinforced with nanoparticles (Rahman et al., 2025b). The integration of nanoparticles enhanced properties such as biodegradability, elasticity, strength, and thermal stability. This method is suitable for achieving the micron thickness of the nanocomposite film (Nguyen-Tri et al., 2018). In this method, liquid colloidal suspension, which is sol, undergoes hydrolysis followed by condensation and produces a gel-like substance. The first step is the formulation of a homogenous solution where polymers are dissolved in suitable solvents (water, ethanol). Nanoparticles

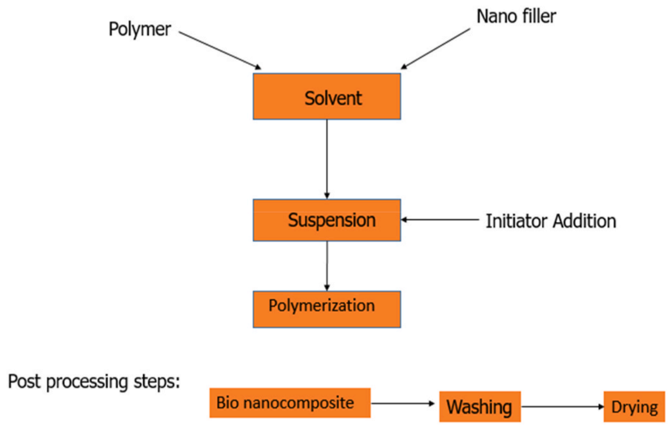



**Fig. 6.** Flow diagram of the manufacturing process of bionanocomposite by the conventional in-situ polymerization technique.

are incorporated into the solution and undergo hydrolysis and condensation reactions; as a result, sol is produced (Pomogailo, 2005). Uniform dispersion is mandatory by stirring or sonication. Newly produced sol is further allowed to go for condensation-specific 3D solid network type of perfect gel type material. Meanwhile, factors affecting (temperature, concentration, $P^{H}$) the gelation reaction and final network structure need to be controlled. Drying is applied to remove excess solvent to obtain nanocomposite material. The sol-gel technique improves the functionality and performance of bio-based polymers by providing precise control over the material's composition and structure at the nano scale. This opens up new possibilities for advancements in biomedical engineering, environmental sustainability, and other fields (Rahman, 2024).

### 3.7. Hot press method

The hot press method is considered the solvent-free prominent fabrication method of the bio-polymeric nanocomposite, where heat and pressure are applied to the mold to make the material physically stronger. This process begins with mixing the material and placing it in a mold where the shape is predetermined. Mold is made from heat-resistant material and is also non-corrosive to any chemical. When the high temperature is applied, polymeric material softens, allowing nanoparticles to be dispersed evenly, resulting in a consolidated structure due to high-pressure compression (Viswanathan et al., 2006). The effects of temperature vary from material to material, like in the case of biopolymers, where a moderate temperature is required to avoid thermal degradation. After the material is subjected to the appropriate pressure and temperature for an adequate amount of time, it is progressively cooled while maintaining its pressure. This creates a robust composite material by locking the nanoparticles and solidifying the polymer matrix. The final nanocomposite is removed from the mold. Commonly employed in various applications, the hot press process is well regarded for yielding dense, consistent, and high-performing composites. It is important to mention that for a better understanding, a schematic flow diagram addressing the hot press method has been shown in Fig. 8.

### 3.8. Melt intercalation

One of the effective techniques for fabrication of polymer-clay nanocomposites is melt intercalation. This approach is totally solvent free what can be expensive and harmful to environment. Selection of appropriate polymer and drying of clay to remove moisture is crucial for enhancing the compatibility. Dried clay is combined with polymer applying heat in an extruder chamber. The strong shear forces generated during extrusion help the clay layers separate, making it easier for the molten polymer to permeate and intercalate between them (Zhang et al., 2017). Temperature, shear rate, and time has considerable effect on the final structure of nanocomposite. Incorporation of cooling solidifies the nanocomposites providing improved qualitative properties. To determine the intercalation and dispersion of clay within the polymer different characterization techniques have been introduced, such as XRD, TEM, SEM etc., (Park et al., 2001). Melt intercalation is an efficient method with suitable diverse applications like wastewater treatment for the selective separation of the toxic pollutants via fixed bed column continuous adsorption process for better findings.

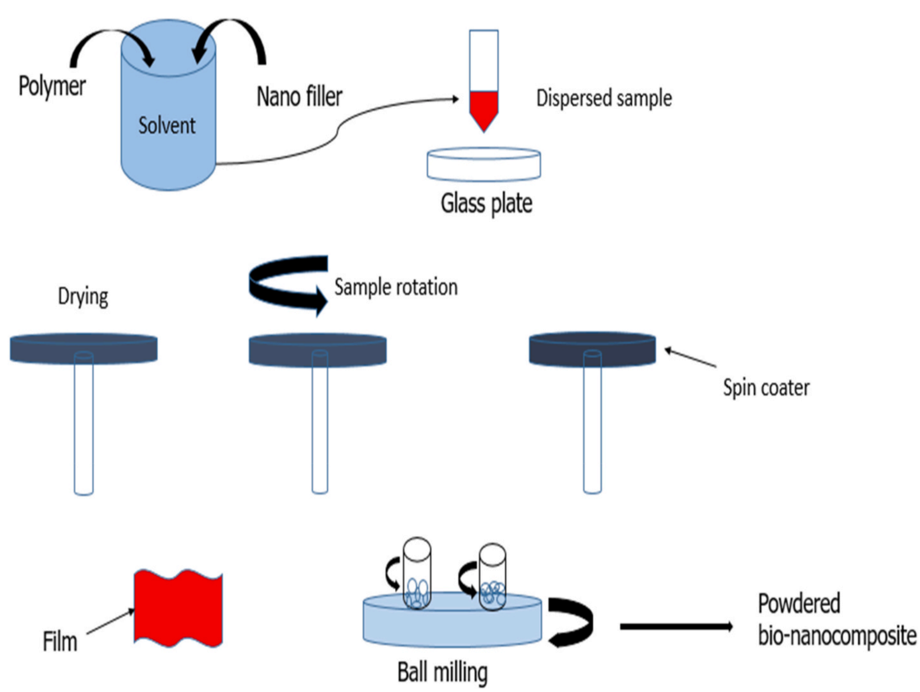



**Fig. 7.** Schematic diagram of bio-nanocomposite manufacturing by the spin coating method.

### 3.9. Cold press method

A good way to create nanocomposite composites for materials that are heat sensitive is through the cold press process. This is simple way to integrate nanoparticles into a polymer matrix without incorporation of high temperature and valuable process where thermal stability is paramount. Depending on desired properties of the final product polymer matrix is chosen basically thermoplastic or thermosets. Nanofiller and polymer are processed into powder by mechanical milling or grinding. Fine powder thoroughly mixed using various process such as ball milling, high shear mixing. Mixing helps to create a homogenous blend where proper dispersion of nanofiller, and polymer occur which is very much critical. After the process mixed material placed into predetermined mold based on desired shape of the final product. Application of high pressure on mold compacts the materials and facilitate interfacial bonding between nanofiller and polymer (Rikhtegar et al., 2017). Curing process may be necessary for chemical crosslinking as a result solidifies the matrix. Proper cooling is mandatory after the curing process for demolded the nanocomposite. Cold press method retains precise control over the composition of nanocomposites. This solvent-free method is environmentally friendly also cuts the production cost while health hazards are in top priority.

### 3.10. Phase inversion method

The phase inversion method is a widely used fabrication technique for producing polymeric membranes and nanocomposite adsorbents, where a homogeneous polymer solution is transformed into a porous solid via phase separation. This technique is particularly effective for designing nanoclay-based nano-adsorbents, owing to its ability to control porosity, surface morphology, and structural uniformity key factors for maximizing adsorption efficiency in bulky industrial wastewater treatment. Advantages of the phase inversion method include simplicity, scalability, and tunable membrane properties. However, disadvantages involve potential challenges in controlling pore interconnectivity and reproducibility under varying environmental conditions. Among the different phase inversion approaches, Evaporation Induced Phase Separation (EIPS) facilitates the formation of dense films by slow solvent evaporation, ideal for adsorbing surface-bound pollutants (Rahman et al., 2024d). Non-solvent Induced Phase Separation (NIPS) involves immersing the polymer/nanoclay solution into a non-solvent bath, rapidly inducing phase separation and forming porous structures with high surface area highly beneficial for removing heavy metals and suspended solids. Thermally Induced Phase Separation (TIPS) relies on temperature-driven demixing of the polymer solution, resulting in membranes with finely tuned pore structures suitable for multi-contaminant adsorption. The incorporation of functionalized nanoclay in these systems enhances the structural integrity, mechanical strength, and adsorption capacity of the final composite, making phase inversion-based nanocomposites a promising solution (Rahman et al., 2025b).

However, in a nutshell, it can be stated that among the fabrication techniques for the development of bio-polymeric nanocomposites, the phase inversion method emerges as the most favorable approach for the eco-friendly, low-cost, and scalable fabrication of clay-based nanosorbent composites. Solution casting and spin coating offer simplicity but suffer from limitations such as long solvent evaporation times, uneven dispersion, and difficulty in controlling internal pore structure. Electrospinning and in-situ polymerization allow for sophisticated material design but often require toxic solvents, expensive equipment, and energy-intensive conditions making them less suitable for large-scale applications. Similarly melt intercalation and hot press methods are solvent-free, but they require high temperatures and shear forces, which may degrade biopolymers and restrict use with heat-sensitive additives like CNCs. While dip coating and sol-gel offer simple or molecular-level fabrication routes, they often result in films with less control over internal porosity or require chemical precursors that may not be environmentally benign. Cold press methods are solvent-free and simple but lack the ability to form porous structures, limiting their utility in adsorption. On the other hand phase inversion particularly EIPS, NIPS,

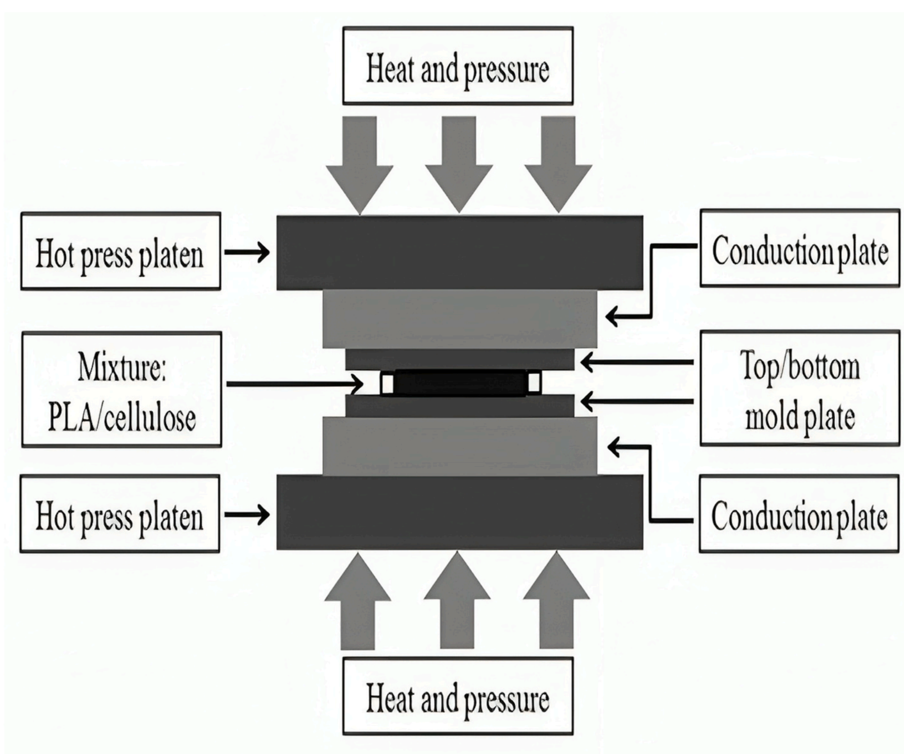



**Fig. 8.** Diagram of hot press method for the fabrication of cellulose-based biopolymeric nanocomposites generated from (Penjumras et al., 2016).

and TIPS provides excellent control over porosity, tunable morphology, and compatibility with water-based or green solvents aligning well with sustainable manufacturing principles. Its relatively simple setup, ambient or moderate operating conditions, and ability to produce mechanically robust, highly adsorptive structures make it especially suitable for functionalized clay-CNC composite fabrication. However, for better understanding the contribution, selectivity, importants, and scope of the selected advance fabrication methods a comparatrive table indication their advantages and disadvantages have been shown in Table 4.

## 4. Characterization process of FC-CNC nanocomposites

### 4.1. X-ray diffraction analysis

X-ray diffraction is a fast and non-destructive analytical technique mostly employed to find the crystalline structure, chemical composition, and crystal grain size of polymeric bionanocomposites being studied (Hassan et al., 2024; Yusof et al., 2019; Madhu et al., 2019; Liu and Hu, 2008). This method enables the analysis of scanned samples across a 2θ range, generally from 5° to 80° (Rahman et al., 2023b). The resulting spectrum is typically obtained from the measurements of the polymeric samples being examined, offering clear diffraction data for both crystalline and amorphous areas. Md. Mahafujul Hassan and his teammates conducted the XRD analysis (Fig. 9a) of untreated, alkaline-treated, bleached SLSF, and CNC reveals structural changes in cellulose crystallinity. The raw SLSF shows a peak at 22.83° (cellulose I), which shifts to 22.14° following alkali treatment, indicating an increase in crystallinity. After bleaching, the peak further shifts to 23.62°, signifying the presence of purified cellulose. CNC displays a sharp peak at 21.93°, which is characteristic of cellulose II resulting from acid hydrolysis. Additionally, smaller peaks indicate the presence of residual ordered structures. The crystallinity index rises from 29.31 % (raw) to 38.34 % (alkali-treated), 62.25 % (bleached), and 81.89 % (CNC), confirming the removal of amorphous regions and an enhancement in the crystalline structure (Hassan et al., 2024). Another example has been shown in Fig. 9b., which deals with the XRD analysis of the CNC-AC bionanosorbents along with their crystal planes and degree of crystallinity (while a = Raw fiber, b = Alkali treated fiber, c = Bleached fiber, d = CNC, e = CNC-AC biopolymeric nanosorbents (Rahman et al., 2024d). Furthermore, the crystallinity index ($C_rI$) can be determined using the following mathematical formula (Rahman et al., 2023a):

$$C_rI = \frac{I_{CA}}{I_{CA} + I_{AM}} \times 100 \tag{2}$$

In this context, $I_{CA}$ refers to the crystalline region, while $I_{AM}$ denotes the amorphous region in the diffractogram of the biopolymeric samples

**Table 4**
Fabrication pathways for functionalized Clay–CNC bio-nanocomposites: A comparative analysis of techniques, advantages, and disadvantages.

| Name of methods | Advantages | Disadvantages |
|---|---|---|
| Solution Casting | 1 Simple and versatile technique.<br>2 Suitable for achieving uniform dispersion of CNC and clay in the polymer matrix.<br>.Can produce films with 3enhanced barrier properties. | 1 Solvent evaporation step is time-consuming, especially for thicker films.<br>2 Requires careful selection of solvents and polymers for compatibility. |
| In-situ polymerization | 1 Ensures uniform distribution of nanoparticles in the polymer matrix.<br>2 Enhances interfacial interaction between components, reducing agglomeration.<br>3 Produces materials with improved thermal stability, mechanical strength, and barrier properties. | 1 Requires precise control of reaction conditions (e.g., temperature, UV light).<br>2 Post-processing (washing, drying) can be complex. |
| Spin coating | 1 Produces uniform, thin films with controlled thickness.<br>2 Fast and efficient for small-scale production.<br>3 Enhances mechanical properties due to reinforcement from nanofillers. | 1 Limited to flat substrates.<br>2 Thickness control depends on solution concentration and spinning speed, which can be tricky to optimize. |
| Electrospinning | 1 Produces nanofibrous mats with high surface area, ideal for adsorption applications.<br>2 Suitable for biomedical and environmental uses (e.g., wastewater treatment).<br>3 Allows fine-tuning of fiber properties by adjusting voltage, viscosity, and distance. | 1 Requires high-voltage equipment and precise parameter control.<br>2 Solvent evaporation must be carefully managed to avoid defects. |
| Dip Coating | 1 Low-cost and simple method.<br>2 Suitable for coating irregularly shaped substrates.<br>3 Produces films with good mechanical strength and chemical stability. | 1 Film thickness depends on withdrawal speed, viscosity, and environmental conditions (humidity, temperature).<br>2 Multiple dips may be needed for thicker coatings. |
| Sol-Gel Method | 1 Precise control over material composition and nanostructure.<br>2 Enhances properties like biodegradability, elasticity, and thermal stability.<br>3 Suitable for producing micron-thick films. | 1 Requires careful control of hydrolysis and condensation reactions (pH, temperature).<br>2 Drying steps can lead to cracking or shrinkage. |
| Hot Press Method | 1 Solvent-free, environmentally friendly.<br>2 Produces dense, high-performance composites with strong mechanical properties.<br>3 Suitable for industrial-scale production | 1 Requires high temperature and pressure, which may degrade heat-sensitive materials.<br>2 Limited to moldable shapes. |
| Melt Intercalation | 1 Solvent-free, reducing environmental and health hazards.<br>2 Efficient for dispersing clay layers in polymers under shear forces.<br>3 Suitable for large-scale production | 1 Requires high temperatures, which may degrade some biopolymers.<br>2 Needs specialized equipment (e.g., extruders) |
| Cold Press Method | 1 Suitable for heat-sensitive materials.<br>2 Solvent-free and cost-effective.<br>3 Retains precise control over nanocomposite composition. | 1 Limited to powdered materials, requiring thorough mixing.<br>2 May require additional curing steps for thermosetting polymers. |
| Phase Inversion Method | 1 Produces highly porous membranes with large surface area ideal for adsorption applications<br>2 Allows precise control over pore size and structure through process parameters<br>3 Compatible with various polymers and nanoparticles (clay/CNC)<br>4 Suitable for heat-sensitive biopolymers as it avoids high temperatures | 1 Requires careful selection and handling of often toxic solvents<br>2 Potential for nanoparticle agglomeration during phase separation<br>3 Produces membranes with relatively low mechanical strength<br>4 Long processing times required for complete solvent removal |

(Segal et al., 1959; Öğretmen et al., 2022). Additionally the crystal grain size (L) can be accurately determined using Scherrer's equation:

$$L = \frac{K\lambda}{BCos\theta} \tag{3}$$

Here, K = 0.89 is Scherrer's constant, B denotes the full width at half maximum of the peak, and k represents the wavelength of the radiation.

### 4.2. Fourier transform infrared analysis

Fourier transform infrared (FTIR) spectroscopy has recently become a popular nondestructive analytical method that offers both qualitative and quantitative insights into polymeric samples. This technique is especially useful for identifying active functional groups and their chemical compositions in the biopolymeric samples being studied, as indicated by the infrared absorption spectrum (Safder et al., 2022; Singh et al., 2022; Rahman et al., 2023a, 2023b; Hassan et al., 2024). The capability of FTIR to deliver such extensive information about polymeric structures and their functional characteristics makes it an essential tool in polymer chemistry and nanocomposite research. By enabling researchers to detect chemical bonding, evaluate molecular interactions, and assess the overall integrity of samples without causing damage, FTIR remains a highly valued method for studying biopolymers, particularly in applications involving chitosan-based nanocomposites (Seki et al., 2013; Hassan et al., 2024; Hato and Ray, 2022). FTIR also allows for the assessment of the purity and concentration of polymeric compounds or complexes, and it excels at identifying unknown substances through intramolecular and intermolecular stretching and bending vibrations. The FTIR spectra from CNC-based nanocomposites generally cover a frequency range of 400–4000 $cm^{-1}$, with a resolution of 5 $cm^{-1}$, providing detailed spectral data for analysis. To achieve accurate results, it is advisable to test 4–5 replicas of each sample during experiments, as this enhances the reliability of the findings (Rahman and

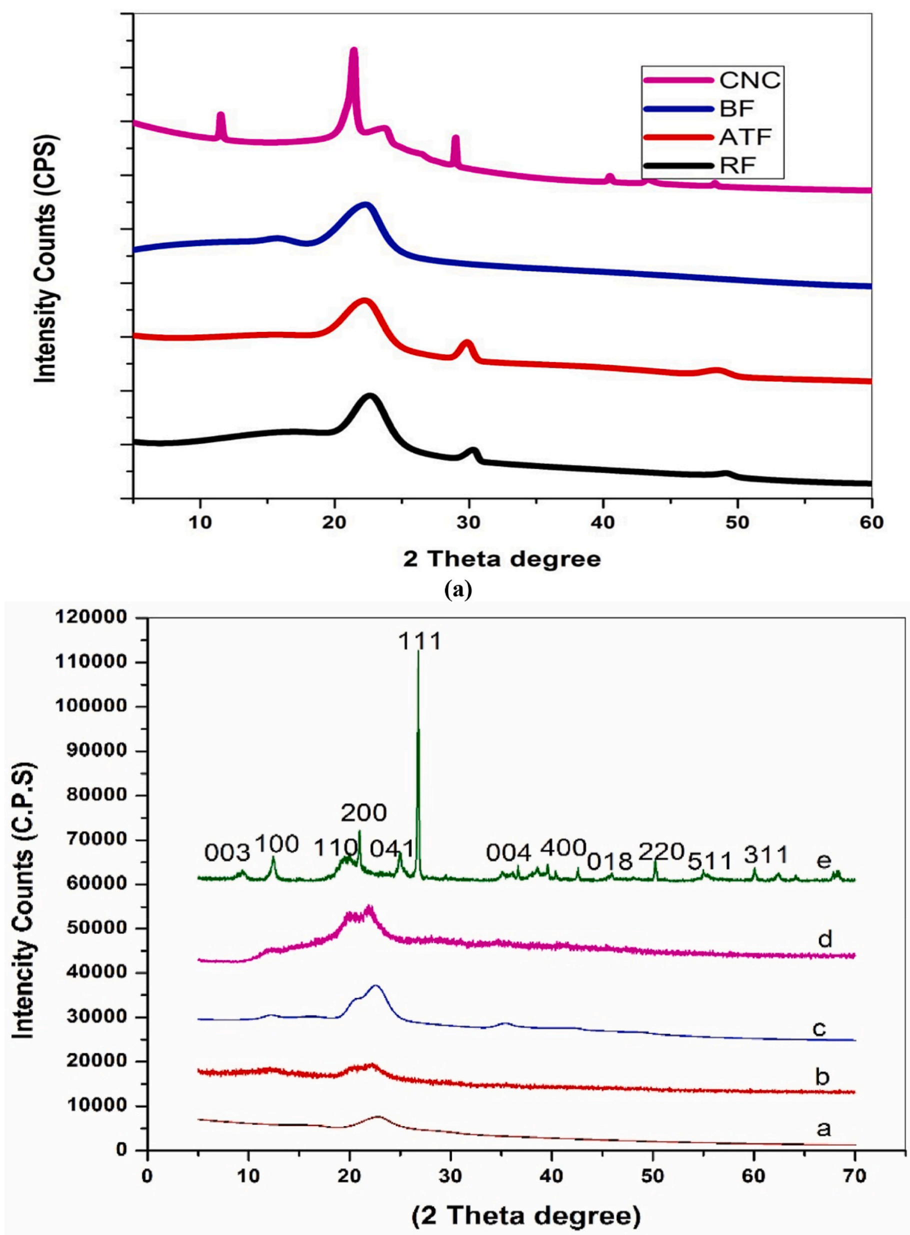
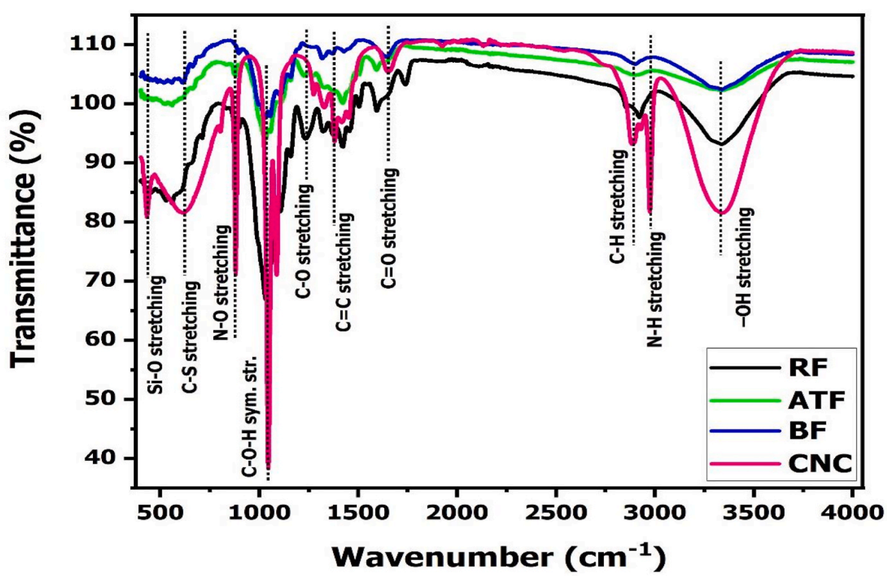



**Fig. 9.** (a) Comparative XRD evaluation of the analyzed raw fiber, alkali-treated fiber, bleached fiber, and CNC from SLSF (Hassan et al., 2024) and **(b)** XRD analysis of CNC-AC bionanosorbents (while a = Raw fiber, b = Alkali treated fiber, c = Bleached fiber, d = CNC, e = CNC-AC biopolymeric nanosorbents (Rahman et al., 2024d). These contents have been regenerated with the permission of Elsevier with license numbers 6001931332764 and 5998590621982.

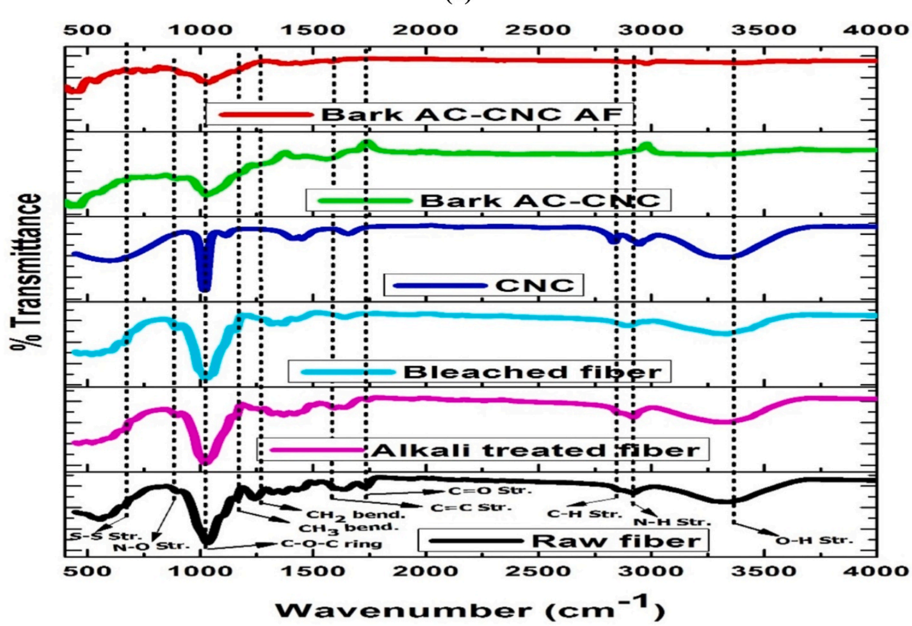
Maniruzzaman, 2021; Madhu et al., 2019; Hassan et al., 2024; Sheikh et al., 2023a,b). Md. Mahmudur Rahman and his co-workers conducted Fourier Transform Infrared-Attenuated Total Reflectance (FTIR-ATR) spectroscopy, which was used to identify the functional groups in CNCs extracted from okra stalks (in Fig. 10a) and banana tree rachis fiber derived CNC-AC based biopolymeric nanoadsorbents (in Fig. 10b). The analysis demonstrated that non-cellulosic components like hemicellulose and lignin were successfully removed through a series of chemical treatments. The CNCs showed characteristic sharpened peaks for hydroxyl (-OH), carbonyl (-COOH), and ether (-C-O-C) groups, confirming the presence of cellulose and effective removal of hemicellulose and lignin. The removal of these unwanted groups enhanced the purity and reactivity of the CNCs, making them suitable for use in bionanocomposite applications (Rahman et al., 2024b, 2024d).

### 4.3. $^{13}C$ cross-polarization magic angle spinning nuclear magnetic resonance analysis

Through the use of carbon-13 solid-state cross-polarization magic angle spinning nuclear magnetic resonance (CP/MAS NMR), a very important tool for the characterization of chitosan-based biopolymeric nanocomposites has been developed that is non-destructive, precise, and efficient (Preston and Forrester, 2004; Rahman and Maniruzzaman, 2023; Pfeffer et al., 1984; Hernandez et al., 1997). This method provides comprehensive insights regarding the molecular structure of biopolymers by concentrating on peak shape, intensity, and chemical shifts, which are the main identifiers of molecular interactions, crystallinity, and chemical compositions (Rahman, 2024; Rahman and Maniruzzaman, 2023; Borchani et al., 2015; Facchinatto et al., 2020). The acquisition of detailed spectra by the CP/MAS NMR allows scientists to




(a)




(b)

**Fig. 10.** (a) The FTIR-ATR spectra comparison of the selected RF, ATF, BF, and CNC extracted from agro-waste biomass, like okra (*Abelmoschus esculentus*) tree sticks (Rahman et al., 2024b) and (b) CNC-AC based biopolymeric nanosorbents (Rahman et al., 2024d). These contents have been regenerated with the permission of Elsevier with license numbers 6001930373295 and 5998590621982.

discover the molecular dynamics and interactions occurring between biopolymeric matrices and their functionalized components. Besides, this technique is the best in determining the crystalline and amorphous regions within the biopolymeric structure by introducing peak deconvolution through the Gaussian or Lorentzian fitting approaches (Liitiä et al., 2003; Atalla and VanderHart, 1999; Park et al., 2009). The approach, which enabled the proper characterization of the structural organization and composition of nanocomposites that are main manipulators for their functional properties, is such an analytical study. Amongst CP/MAS NMR's most recent successes and strengths over the past studies on instrumental techniques is its effectiveness in structural variations, which enhances understanding of biopolymeric materials and promotes industrialization of nanocomposites (Magnani et al., 2022; Rahman, 2024). The exposure to the method's capability of generating accurate and consistent data further enables the development of advanced biopolymeric nanocomposites for various challenging applications, for example, industrial wastewater treatment (Yanat et al., 2023). By supplying hidden information that is key to the material's structural and chemical properties, CP/MAS NMR is still a very useful tool for the performance and sustainability improvement of biopolymeric nanocomposites.

### 4.4. Thermogravimetric analysis

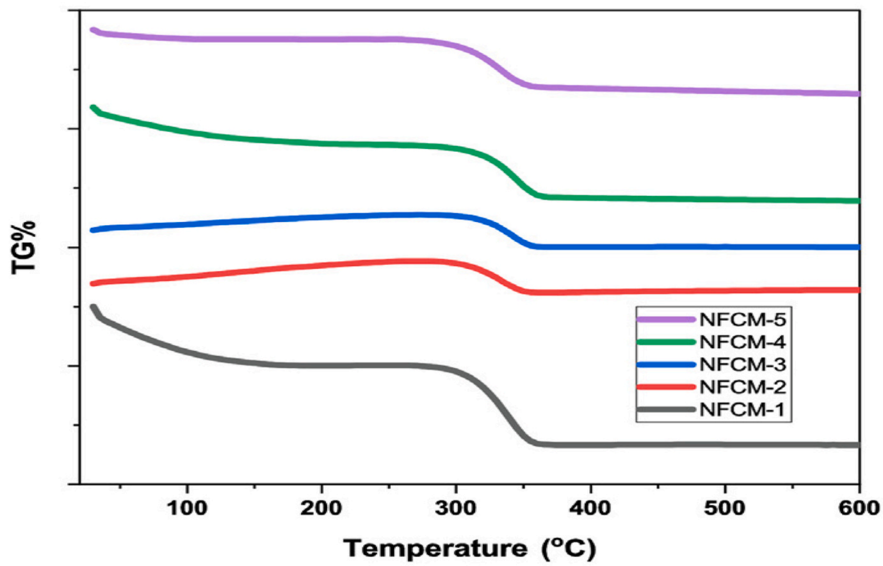
Thermogravimetric analysis (TGA) is a commonly used method for examining the thermal behavior of chitosan-based biopolymeric nanocomposites by tracking mass loss during thermal degradation or decomposition as heat is applied. This technique is essential for gaining insights into the thermal profile of these materials. TGA provides important information regarding the stability, degradation temperatures, and thermal properties of biopolymeric nanocomposites, which are vital for assessing their suitability for different applications (Hassan et al., 2024). Additionally, TGA provides insight into the physical and chemical characteristics, including the chemical composition of the biopolymeric nanocomposites. The data obtained from TGA can also help estimate the activation energy needed for thermal degradation, offering a deeper understanding of the material's thermal stability (Rodrigues et al., 2020; Javaid et al., 2018). To achieve accurate results, the heat applied during the analysis is typically maintained at a constant rate, often reaching up to 1000 °C. Samples are usually placed in an inert atmosphere, such as nitrogen ($N_2$), which acts as a purge gas to avoid unwanted reactions with oxygen. The flow rate of $N_2$ is generally controlled at around 50 mL/min to ensure a stable environment for the analysis. The heating rate, a key factor in TGA, is typically set between 10 and 20 °C per minute. This controlled heating process allows for precise observation of the sample's thermal transitions, including the onset and completion points of degradation. Md. Shamim Sheikh and his teammates investigated and reported the thermal stability of the fabricated GO-CNC membranes was assessed using TGA technique, as illustrated in Fig. 11**a**. The decomposition patterns across various compositions—from NFCM-1 (20 % GO-80 % CNC) to NFCM-5 (60 % GO-40 % CNC)—exhibit a single-step degradation process. Incorporating CNC improves the thermal stability of GO, with a steady increase in stability corresponding to higher GO content. The most significant enhancement in thermal stability is noted in NFCM-4 (50 % GO-50 % CNC). This improvement is linked to the interaction between GO and CNC, which mitigates thermal degradation and bolsters the overall structural integrity of the composite (Sheikh et al., 2023a,b). Another example has been shown in Fig. 11b, which was reported by Rahman and coworkers. They conducted their experiment on the newly fabricated CNC-AC based multifunctional super active biopolymeric nanosorbents, which was used to purify the real-time bulky industrial wastewater for the selective separation of the targeted hazardous toxic pollutants like heavy metals and dyes from water bodies via fixed-bed continuous column adsorption study (Rahman et al., 2024d). Whereas it is noteworthy that the finally fabricated CNC-AC biopolymeric composites have shown the far grater thermal stability up to 700 °C. This could be happened due to the successful incorporation of the mineral rich nanofiller like activated carbon during the experimental session (shown in Fig. 12a-j).

### 4.5. Differential scanning calorimetry analysis

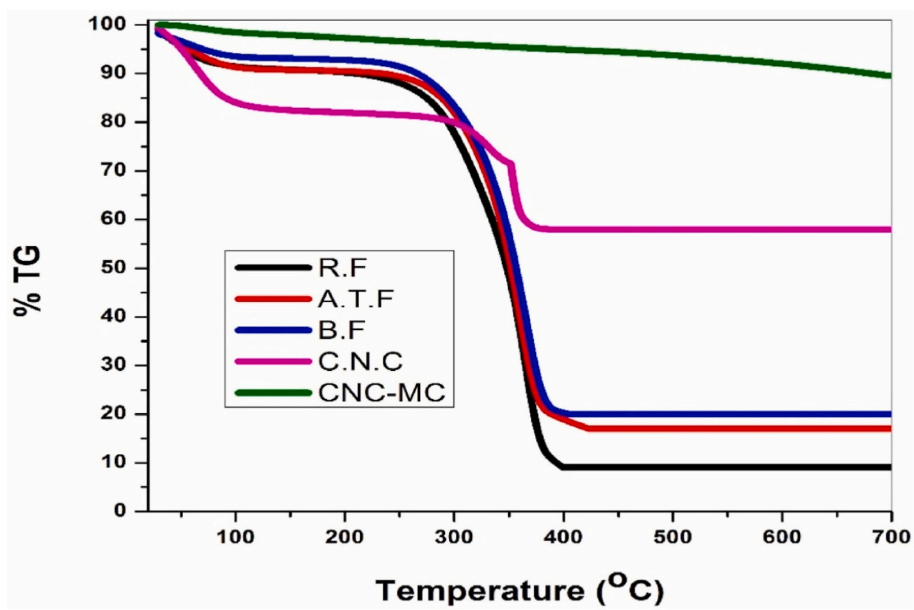
Differential Scanning Calorimetry (DSC) is an excellent way to do thermophysical characterization of functionalized clay cellulose nanocrystal (CNC)-based biopolymeric nanocomposites (Pielichowski and Pielichowska, 2018; Leszczynska and Pielichowski, 2008). The method measures the heat flow associated with the thermal transitions of such temperatures as glass transition temperature ($T_g$), melting temperature ($T_m$) and crystallization temperature ($T_c$), this information is very important for knowing how the material behaves and how it will perform under thermal stress (Corcione and Frigione, 2012). The incorporation of functionalized clay and CNC in the composites increases the thermal performance of the polymer matrix by increasing its Tg and thermal stability. This innovation is due to the strong interfacial interactions between polymer and fillers, like hydrogen bonding and van der Waals forces, which constrain the polymer chain movement (Essomba et al., 2022; Fan et al., 2017). These bonds are vital in wastewater treatment where the materials have to survive high temperature fluctuations. The aspect ratio and the heat transfer barrier of functionalized clay, together with the high crystallinity of CNC, enable it to serve as a structure reinforcement (Das et al., 2020; Hnamte and Pulikkal, 2022). DSC also examines the degree of crystallinity in the polymer matrix that is connected with mechanical strength and lower permeability that makes the nanocomposite more durable (Fan et al., 2017; Das et al., 2020; Yaseen et al., 2024). In addition, DSC is also utilized in determining how well the fillers are dispersed in the matrix. Homogenized dispersion yields the more advantageous thermal properties as evidenced by the definite DSC curves which show shifted or broadened peaks (Akharame et al., 2018). The thermal cycling stability of the material, which is evaluated by DSC, makes it possible for the material to be reliable in long-term demanding industrial environments (Heydari et al., 2017). In the whole area of analysis, DSC is the centerpiece that is required for optimizing the thermal properties and structural integrity in these nanocomposites, which makes them the best candidates for industrial wastewater treatment. With its capability to enhance the material's thermal stability and functionality, DSC supports the development of sustainable and high-performance solutions for tackling complex environmental issues in effluent management systems (Hnamte and Pulikkal, 2022; Yaseen et al., 2024).

### 4.6. Field emission scanning electron microscope analysis

Recent studies have extensively examined the surface microstructure of chitosan-based biopolymeric nanocomposite samples using the field emission scanning electron microscope (FESEM) technique. To improve imaging quality, these samples are usually mounted on aluminum stubs with carbon tape and then coated with a thin layer of gold, platinum, or carbon, typically around 5 nm thick. This coating enhances the sample's conductivity, resulting in clearer and higher-quality micrographs. Besides capturing detailed images, the FESEM allows for the observation of surface morphology and topography, highlighting the microstructural features of the biopolymeric samples (Liu et al., 2021a,b; Hassan et al., 2024; Williams, 2021; Egerton, 2005). This method enables researchers to accurately evaluate various aspects of the surface architecture, such as particle size and distribution. These measurements are further refined with specialized software like ImageJ and OriginLab, which facilitate precise particle size analysis and the generation of size distribution curves (Sheikh et al., 2023a,b). The combination of FESEM imaging and software analysis offers a thorough understanding of the microstructure and surface characteristics of chitosan-based biopolymeric nanocomposites, making it a popular technique for detailed surface




(a)




(b)

**Fig. 11.** (a) TGA analysis of various CNC-based biopolymeric nanocomposite membranes (Sheikh et al., 2023a,b) and (b) TGA analysis of the fabricated CNC-MC bionanosorbents (Rahman et al., 2024a). These contents were regenerated with permission from Elsevier with license numbers 5997470877661 and 5998600199924.

characterization in materials research. Md. Shamim Sheikh and his teammates explores the properties of Graphene Oxide-Cellulose Nanocrystal (GO-CNC) composite membranes, particularly how CNC disperses within the GO matrix and the effects on the membrane's characteristics. FESEM analysis shows that GO tends to be oriented or trapped on the surface of CNC, which minimizes intercellular gaps and creates smoother surfaces. When CNC content is low, nano-cellulose integrates well into the GO matrix, but at higher concentrations, agglomeration occurs, negatively impacting the properties of the composite. The microvoid surfaces of GO-CNC composites exhibit greater homogeneity compared to pure CNC samples, suggesting a strong interaction and effective blending of GO and CNC in the composite matrix (Sheikh et al., 2023a,b).

### 4.7. *Transmission electron microscope analysis*

The transmission electron microscope (TEM) is one of the most advanced and widely used techniques for obtaining detailed information about samples, revealing their 3D structure, surface morphology, surface topography, and chemical composition, including the characteristics of chemical bonds within the sample's structure (Choudhary et al., 2019; Bodnar et al., 2005; Saka, 2003). However, a significant challenge in using TEM for analyzing nanopolymeric materials has been the preparation of thin foil samples. To tackle this issue, a novel method called focused ion beam (FIB) technology has recently gained attention (Reza et al., 2015; Liu et al., 2009). This technique allows TEM micrographs to provide more accurate measurements, especially when determining the diameter of exposed biopolymeric samples. FIB can detect even the smallest details, such as the transverse dimensions of

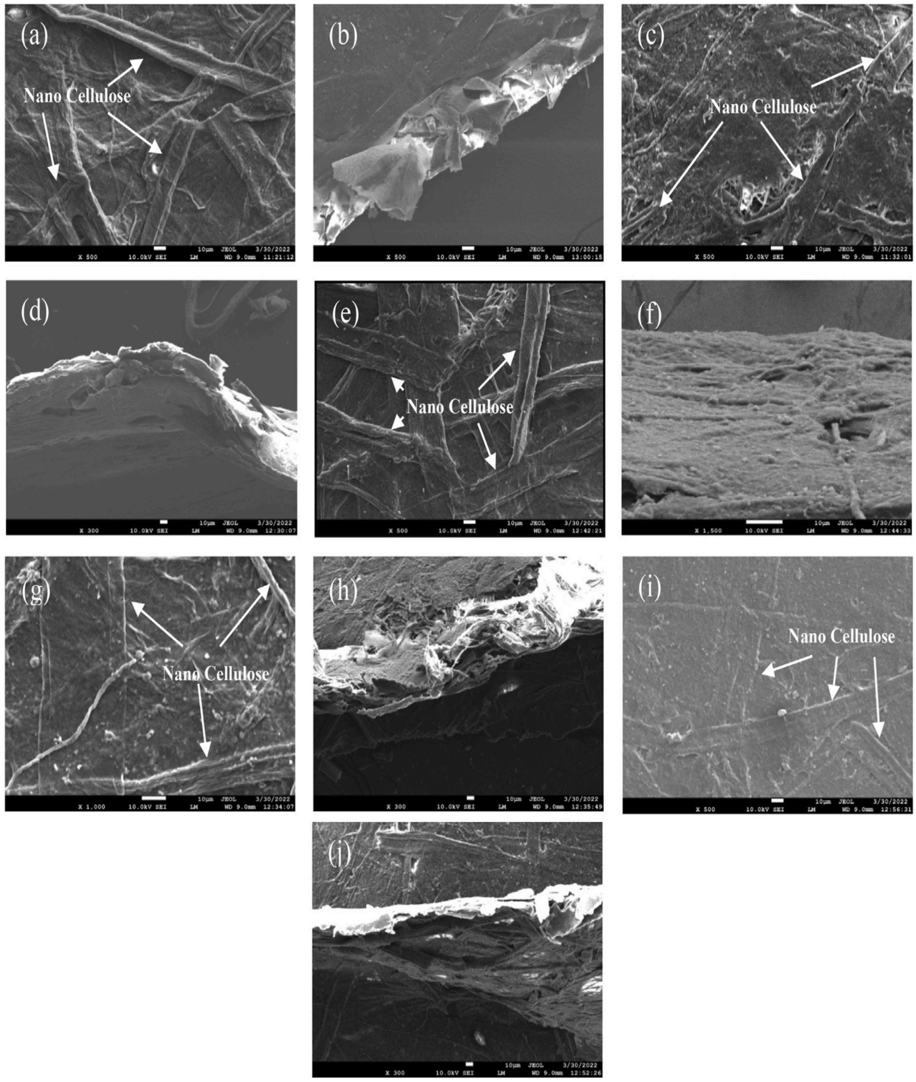



**Fig. 12.** FESEM analysis of the surface and cross-sectional areas: (a) & (b) for NFCM-1, (c) & (d) for NFCM-2, (e) & (f) for NFCM-3, (g) & (h) for NFCM-4, and (i) & (j) for NFCM-5 (Sheikh et al., 2023a,b) and this content regenerated with the permission from Elsevier with a license number 5997470877661.

various sublayers, which are essential for understanding the construction of single-cell walls in polysaccharides. This advancement has greatly enhanced the accuracy of TEM analysis, offering deeper insights into the minute structures of nanopolymeric materials and improving the overall exploration of their properties. The combination of TEM and FIB enables researchers to capture intricate details of biopolymeric samples with much greater precision, making it an invaluable tool in the field of material science (Beakou et al., 2008; Manimaran et al., 2020).

### *4.8. Atomic force microscopy analysis*

Atomic force microscopy (AFM) is an effective technique for examining the surface microstructural topography of chitosan-based biopolymeric nanocomposite specimens with remarkable resolution, reaching the nanometer or even sub-nanometer scale. This method is increasingly popular in modern research, particularly in wide-range contact mode. The process involves scanning images with a scanning near-field optical microscopy (SNOM) system, which features unique, high-quality microfabricated sensors, such as a silicon cantilever topped with a hollow pyramid tip. During scanning, the rate is typically set to about 3 lines per second, while the resonance frequency ranges from 210 to 490 kHz (Rahman and Maniruzzaman, 2023; Fauzi et al., 2020; Senthamaraikannan et al., 2016). The images captured have a resolution of approximately $2048 \times 2048$ pixels. The roughness of these images is assessed using the root mean square (RMS) value, which reflects the height variation from the mean plane of the surface, allowing for an accurate representation of the topography. Subsequently, the images are aligned with the mean plane to minimize discrepancies. This advanced technique not only provides a detailed 3D representation of the sample's surface but also allows for precise measurement of the forces involved in adhesion behavior (Ali and Aldujaili, 2022; Rahman et al., 2024). AFM can deliver comprehensive insights into the surface-oriented topographic features of the samples, including their three-dimensional structure. Additionally, the technique enables highly accurate measurements of the diameter of the chitosan-based biopolymeric specimens. This multifunctional method is well-regarded for its precision in both imaging and force measurement, making it a key tool for investigating the surface and structural characteristics of nanocomposites.

### *4.9. X-ray photoelectron spectroscopy*

X-ray photoelectron spectroscopy (XPS) is truly a well-known and frequently applied technique to analyze solid-state materials. It utilizes the photoelectric effect discovered by Einstein which is by recording the kinetic energy of electrons that have been released from a material when irradiated with X-rays (Hüfner, 2013). The energy of these emitted core electrons is specific to the elements, thus enabling the determination of the elements and the characterization of their chemical bonds (Hüfner, 2013; Powell, 1988; Hagström et al., 1964). XPS - the name of the technique that is commonly used for chemical analysis is electron spectroscopy. Because of the scattered photoelectrons and other electrons in the material, XPS limits the emitted photoelectrons to a short distance, usually just a few nanometers, thereby only the surface of the material is analyzed (Seah and Dench, 1979; Baschenko and Nefedov, 1979). For biopolymeric nanocomposites made from functionalized clay and cellulose nanocrystals (CNC), XPS is very important for understanding and improving their performance in treating industrial wastewater. Thus, the surface will be illuminated with X-rays and the kinetic energies of the emitted photoelectrons will be measured in order to determine the elemental composition of the material (Son et al., 2020; Sahoo et al., 2009). Consequently, this procedure acts as a window to a particular element, functional groups that have been identified and chemical interactions that are proven out. In these nanocomposites, XPS verifies that the clay and CNC have been successfully chemically functionalized, using hydroxyl, carboxyl, or amine groups (Badruddoza et al., 2013; Samai and Bhattacharya, 2018). The effectiveness of the treatment is increased by these groups, which possess greater adsorption capacity. These functional groups bind the pollutants, such as heavy metals, dyes, and organic compounds, which are present in industrial wastewater (Rahman and Maniruzzaman, 2021; Pan et al., 2010). XPS helps to identify the nature of interfacial interactions between fillers (clay and CNC) and the biopolymeric matrix. The formation of various types of bonds such as covalent or hydrogen bonds, which provide the foundation for weakly bonded monomers and their subsequent alignment into polystyrene chains, is the trigger for both the uniformity and compatibility of the polymeric nanocomposite that consequently lead to improved mechanical and thermal behavior (Saleh et al., 2019; Akharame et al., 2018; Pan et al., 2010). XPS evidenced the surface changes including the presence of oxygen- or nitrogen-containing groups, indicating the increased hydrophilicity and reactivity of the material, thereby improving its performance as an adsorbent (Saleh et al., 2019; Akharame et al., 2018). The analysis, thus, also facilitates the material's chemical stability check by delineating the possible changes of functional groups caused by the effluents from industries. Additionally, XPS offers depth profiling, enabling researchers to study how elements are spread across the surface and deeper layers of the material (Musico et al., 2013). This is important for understanding how well the nanocomposite holds up under real-world conditions. By giving detailed information about the surface, XPS helps create better composites with specific properties that work well for treating wastewater (Pan et al., 2010). Overall, XPS is a crucial tool for improving the design of nanocomposites made from functionalized clay CNC, ensuring they have the right surface features, chemical stability, and performance to handle large amounts of industrial wastewater.

### 4.10. Brunauer-Emmett-Teller analysis

Brunauer-Emmett-Teller (BET) analysis is a crucial technique in determining the surface area, pore size, and porosity of functionalized clay cellulose nanocrystal (CNC)-based biopolymeric nanocomposites that are important for the industrial wastewater treatment application (Mohan et al., 2020; Lai et al., 2017; Kamal et al., 2017). BET is a technique that evaluates the adsorption of gases, usually nitrogen, on the material's surface, and thus these parameters are determined that are directly related to the adsorption capacity and pollutant removal efficiency of the composite (Lofrano et al., 2016). Functionalized materials such as clay and CNC are responsible for the high surface area and the incorporation of micro- and mesopores, which are active sites for the retention of hazardous metals and dyes (Lofrano et al., 2016; Shabtai et al., 2021). This high porosity and surface area are the reasons for the better transport of mass, which allows the pollutants to be adsorbed in a faster and more efficient way. The functionalization of the fillers with groups such as hydroxyl or amine also contributes to the increased adsorption by making more active sites available and allowing the contaminants to interact with the sites (Muslim et al., 2023; Chong et al., 2015). BET analysis also serves in determining the dispersion of fillers within the polymer matrix, as bad dispersion can decrease the effective surface area (Muslim et al., 2023). For wastewater treatment, compounds of large surface area and optimal pore size dispersions are the main pillars of large pollutant loadings and operational durability, respectively (Al-Hetlani et al., 2021). BET analysis further facilitates the assessment of the composite's stability after several cycles of use, thus ensuring its reusability and sustainability.

### 4.11. Wastewater analysis

Atomic Absorption Spectroscopy (AAS) has been a staple in detecting metallic and heavy metal ions such as Cu, Ni, Co, Cd, Zn, Mn, Pb, Cr, Hg, As, Be, Al, Fe, Ca, and Mg in contaminated water, relying on the absorption of light by gaseous atoms at parts per million (ppm) to parts per billion (ppb) levels (Assubaie, 2015; Wysocka, 2021). However, this method has its drawbacks, including limited sensitivity and the capability to analyze only one element at a time. It also faces challenges in accurately measuring certain elements like Mo and Si, which tend to form oxides in the flame of the hollow cathode lamp. To overcome these limitations, a more sophisticated technique called inductively coupled plasma mass spectrometry (ICP-MS) has become the preferred choice. ICP-MS is extensively utilized for detecting metallic and heavy metal ions in various aqueous environments, spanning fields such as soil and earth sciences, environmental and life sciences, forensic science, and large-scale industrial applications (Wysocka, 2021). The technique's effectiveness is attributed to the high ion density and temperature generated in the plasma system of the inductively coupled plasma torch, making it an excellent atomizer and ionizer for a wide range of sample types and matrices. Its remarkable characteristics, including high sensitivity (ppb–ppq levels), salt tolerance, independent elemental responses, and exceptional accuracy and precision in quantification, establish ICP-MS as a highly dependable tool for detecting and quantifying trace elements (El Messaoudi et al., 2024a,b; Sheikh et al., 2023). In addition to ICP-MS, ultraviolet-near infrared (UV-NIR) spectroscopy has also proven effective in detecting both organic and inorganic dyes (Guan et al., 2018). This method functions by absorbing specific wavelengths of light in the ultraviolet and near-infrared regions, in accordance with the Beer-Lambert law (Li et al., 2020). Furthermore, various types of instrumentation have been utilized to identify other emerging contaminants. For example, pesticide residues can be detected using techniques like gas chromatography-mass spectrometry (GC-MS) or gas chromatography-tandem mass spectrometry (GC-MS/MS) (Chan et al., 2022). In contrast, antibiotic residues are typically identified through high-performance liquid chromatography (HPLC), liquid chromatography-mass spectrometry (LC-MS), or liquid chromatography-tandem mass spectrometry (LC-MS/MS) (Tasci et al., 2021; Othman et al., 2023). Additionally, methods such as Raman spectroscopy, optical microscopy, and Fourier transform infrared (FTIR) spectroscopy have proven effective in identifying microplastics and nanoplastics, showcasing the adaptability of these sophisticated analytical techniques in tackling intricate environmental contamination challenges (Jin et al., 2022; Chakraborty et al., 2023).

## 5. Application of the fabricated FC-CNC bionanoadsorbents in wastewater treatment technology

A natural clay material consists of minerals and organic particles that possess polyfunctional properties due to its well-organized crystal structure and hydrophilic nature. There are several types of clay that comprise a variety of $[SiO_4]^{4-}$ (tetrahedral) and $[AlO_3(OH)_3]^{6-}$ (octahedral) randomly oriented structures, connected through weak van der Waals forces and containing many positively charged metallic ions

(Rahman et al., 2023; Rahman, 2024). Elsewhere, CNC is a multifunctional biopolysaccharide that reveals high porosity, an extended surface area, and negatively charged functional groups, which impart effective functionality in industrial water treatment when combined with highly porous clay materials (Dewan et al., 2025; Rabby et al., 2025). Several physical, chemical, or mechanical techniques/methods are involved in forming composite materials, where blending, shear force, ball milling, and curing could be instrumental in achieving desired properties through fabrication. Therefore, a highly active, bulky surface area, chemically reactive, high mechanical strength (aiding in reuse several times), extended porosity, and permeable CNC-Clay composite have been developed, which is appropriate for water treatment technology as an adsorbent in specific forms such as beads, membranes, and powders, as shown in Fig. 13. The composite forms suitable bonds through the negatively charged functional group of CNC particles (-OH, -$NH_2$, etc.) that convey lone pair electrons (results chemisorption) and the positively charged functional groups of clay particles via electrostatic interactions, hydrogen bonding, and cation exchange, which makes it effective against the targeted ionic pollutants (e.g., Pb, Cd, Hg, As, etc.) as an adsorbent, where clay helps to bind metals through ion exchange, interparticular diffusion, deep penetration, physisorption while CNC provides additional active sites. Although there have been several adsorption techniques applied in industries to treat water, nevertheless two types of adsorption techniques are practiced: (i) fixed bed column and (ii) batch adsorption, where fixed bed column adsorption is more desired considering suitable cost and process optimization (Rahman et al., 2024h), as shown in Fig. 14. An effective comparison between batch adsorption and fixed bed continuous column adsorption study has been tabulated below in Table 5, considering the real-time water treatment. The CNC-clay-based composite can also be effective in the removal of organic pollutants (e.g., phenols, solvents, aromatic hydrocarbons, polychlorinated biphenyls, etc.) and textile dyes (e.g., methylene blue, methyl orange, Congo red, and vat dyes). Besides, it imparts benefits as a filtration membrane to eliminate bacteria, viruses, and particulates by improving water purity. The composite can also be applied as catalyst support (while degrading organic pollutants under sunlight, the composite acts as a support for the catalyst), and for pH regulation (providing support to maintain pH levels) and nutrient regulation (assisting in adsorbing additional nutrients in water) in industrial water treatment technology. The synthesis methods are compatible with existing manufacturing infrastructure, further easing the path to commercialization. Another benefit is the robust mechanical and chemical stability of the FC-CNC composite. Multiple studies confirm its ability to withstand repeated adsorption-desorption cycles with minimal loss of performance. Simple regeneration using acid or alkaline washing has proven effective for up to five or more cycles, improving both cost-efficiency and material longevity. This reusability aligns well with circular economy principles by reducing waste and lowering operational costs. Certain technical challenges must be managed during scale-up including risk of clogging in continuous-flow systems, which can be mitigated by incorporating pre-filtration steps to remove suspended solids. Process optimization studies suggest that maintaining a column bed height between 50 and 100 cm strikes an effective balance between adsorption efficiency and pressure drop. Maintaining appropriate operating pH and temperature is critical, as these parameters strongly influence adsorption performance. The environmental implications of using FC-CNC materials are also favorable. As these composites are primarily derived from renewable sources (e.g., agricultural biomass for CNC and naturally occurring clays), they offer a sustainable and biodegradable alternative to conventional synthetic adsorbents. Consequently, this eco-friendly water treatment solutions can be desired to achieve significant effectiveness in generating a green environment, sustainable development, and circular economy practices.

## 6. Mathematical models

Several mathematical models have been implemented for optimizing and designing data obtained from continuous column processes, such as the Yoon-Nelson, Clark, Thomas, Bed depth service time, Modified dose response, Adams and Bohart model, Wolborska and Yan models for correlation with the breakthrough curves (Rahman et al., 2024a). Besides the pseudo-first-order and pseudo-second-order kinetics, the Dubinin-Astakhov isotherm and the Freundlich and Langmuir isotherms are also applied in batch adsorption depending on the system. The Breakthrough Curve (BTC) is the most prominent for wastewater treatment analysis when an appropriate design of the column is needed for better precision of the effluent treatment. It is obtained by the final to initial (adsorbent/toxicant) wastewater solution concentration ($C_f/C_i$) ratio versus conduction time t (in minutes), which is significant in mathematical correlations for the data obtained from continuous column adsorption studies. Some crucial factors, such as intraparticle diffusion, film diffusion, axial dispersion, nonlinear isotherm, and external mass transfer, need to be considered for developing a detailed model to evaluate efficiency and large-scale implementations (Rahman, 2024). Some developed mathematical models indicating linear and non-linear equations used widely are described below. Several practical

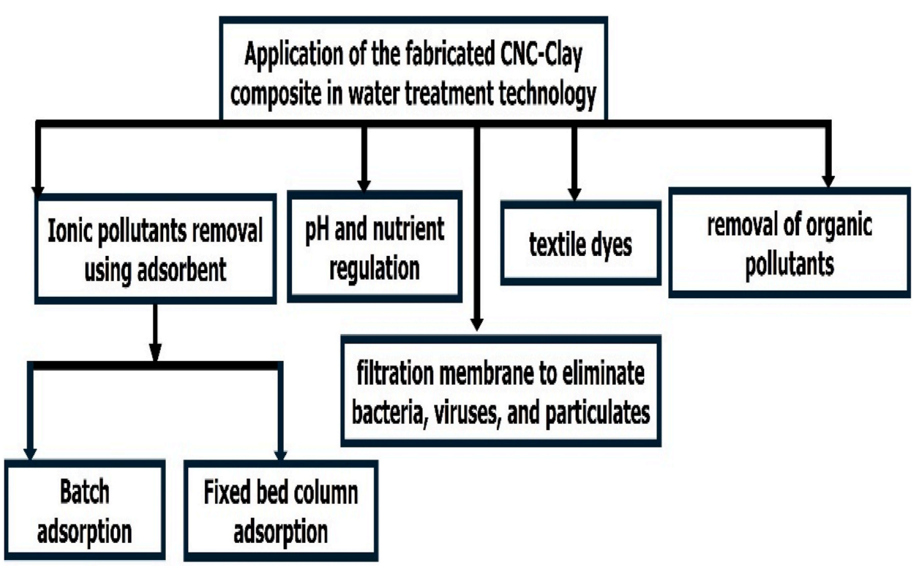



**Fig. 13.** Different applications of fabricated CNC-clay composite in water treatment technology.

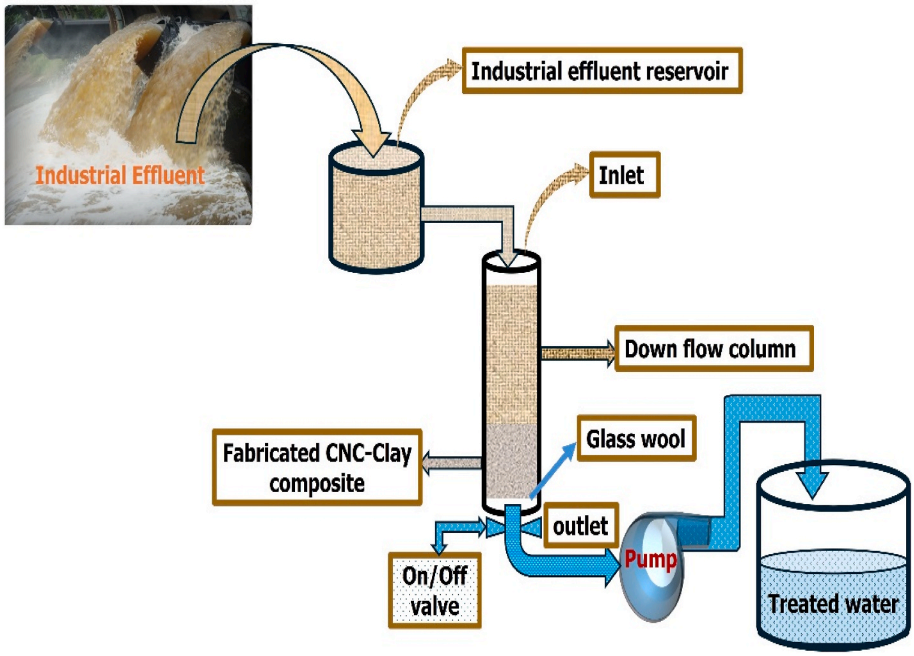



**Fig. 14.** An overview of a fixed bed downflow column, which can be applied at the lab scale for process optimization through a fabricated CNC-clay composite adsorbent.

features (regeneration time, capacity, operating lifespan, etc.) and the efficiency of the column can be analyzed using mathematical modeling. The Yoon-Nelson, Clark, Thomas, Bed depth service time, Adams and Bohart model, Modified dose response, and Wolborska models are mostly used for theoretical assumptions, using data obtained from vertical fixed bed column adsorption studies with varying flow rates and concentrations. These models serve several critical purposes, allowing researchers to estimate breakthrough time, determine maximum adsorption capacity, evaluate bed exhaustion rates, and predict how the system will behave under varying operating conditions (e.g., flow rate, bed height, or influent concentration). These models are essential tools in scaling up from pilot-scale data or laboratory to industrial treatment systems. They reduce uncertainty in system design and operation, support cost-benefit assessments, and ensure that treatment systems meet regulatory discharge standards. However, for better clarity, a non-lonier curve fitting of the fixed-bed continuous column adsorption models, including the Yoon-Nelson, Thomas, Clark, Adam-Bohart, and Wolborska model, to the experimental BTC (Breakthrough Curve) for eliminating the particular toxicants like both the heavy metals and dyes from the wastewater bodies have been shown in Fig. 15 according to the study of (Rahman et al., 2024a).

### 6.1. The Yoon-Nelson model (YSM)

The Yoon-Nelson model assumes that the decreasing adsorption rate is directly proportional to the adsorbate adsorption and 50 % breakthrough on the adsorbent, which doesn't emphasize the adsorbate properties, any adsorbent types, and other corporal features of the adsorption bed (Rahman et al., 2023d). The linear and non-linear equations are labeled below:

$$ln\left(\frac{C_t}{C_0 - C_t}\right) = K_{YN}\,(\tau - t) \quad (4)\ (\text{linear})$$

$$\text{and } \frac{C_t}{C_0} = \frac{1}{1 + \exp[K_{YN}\,(\tau - t)]} \quad (5)\ (\text{non-linear})$$

Where, $K_{YN}$, and $\tau$ implies the rate constant ($min^{-1}$), and 50 % breakthrough time (min) that is obtained from ln ($\frac{C_t}{C_t}$ vs t) intercept and slope. Hence, t, $C_0$, and $C_t$ is the time (min), adsorbate initial concentration (ppm), and final concentration (ppm) respectively.

### 6.2. The Thomas model

The Thomas model is the general and most widely used model for column studies, which performs well in the absence of certain limitations, i.e., external and internal diffusion. This model follows rate-driving Langmuir isotherm and second-order reversible reaction kinetics for adsorption (Rahman et al., 2023a). The linearized and non-linearized expression are labeled by the following equation:

$$ln\left[\left(\frac{C_o}{C_t}\right)^{n-1} - 1\right] = \left[\left(\frac{K_{Th} q_o m}{Q}\right) - K_{Th} C_{-}(o)\ t\right] \quad (6)\ (\text{Linear})$$

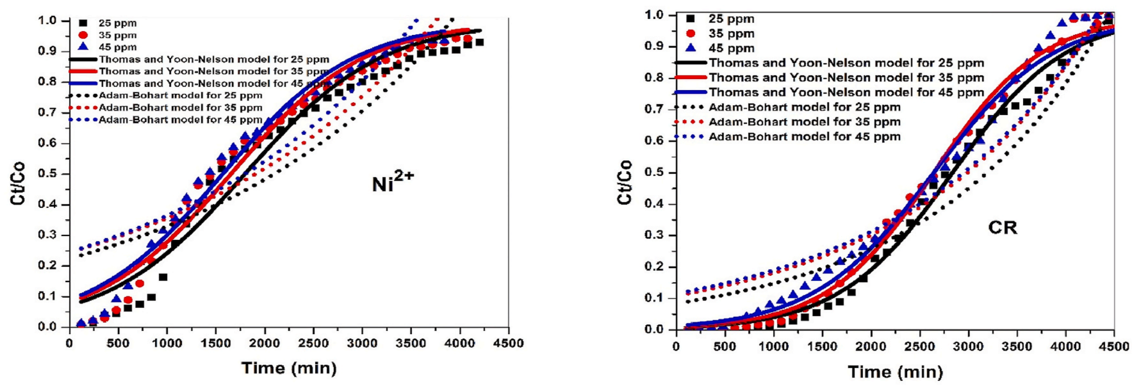
$$\text{and } \frac{C_t}{C_0} = \frac{1}{1 + \exp[K_{TH}\,(\,q_0 m - C_0 V)]} \quad (7)\ (\text{Non-linear})$$

Where, $K_{Th}$, and $q_o$ implies Thomas rate constant ($mL\ min^{-1}\ mg^{-1}$), and maximum solid phase concentration (mg/g) that is obtained from $ln\left[\left(\frac{C_o}{C_t}\right)\right.$- 1] vs t intercept and slope. Hence, t, $C_0$, and $C_t$ is the time (min), adsorbate initial concentration (ppm), and final concentration (ppm) respectively.

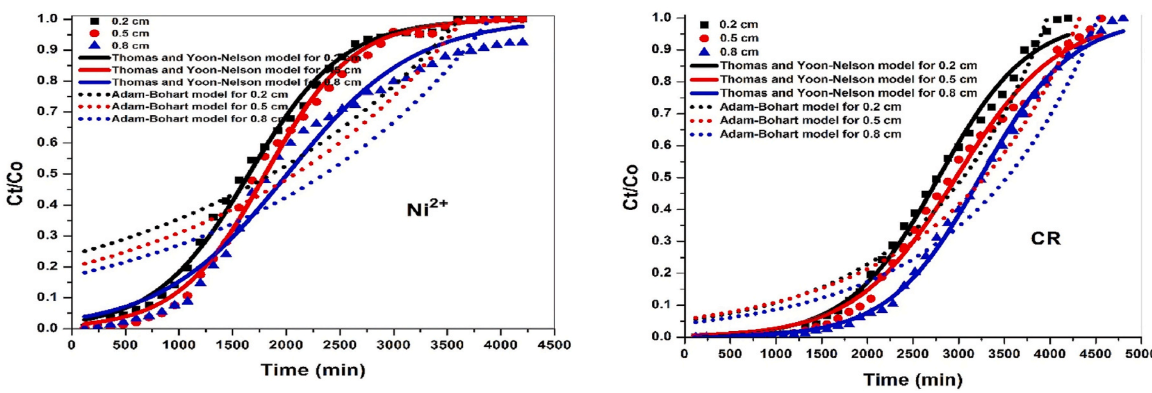
### 6.3. Clark model

The Clark model is developed for breakthrough curves to the intended use of the mass transfer concept (solved system equations) in combination with the Freundlich isotherm by neglecting dispersion phenomena, which is expressed by the following linear and non-linear equation (Patel, 2019):

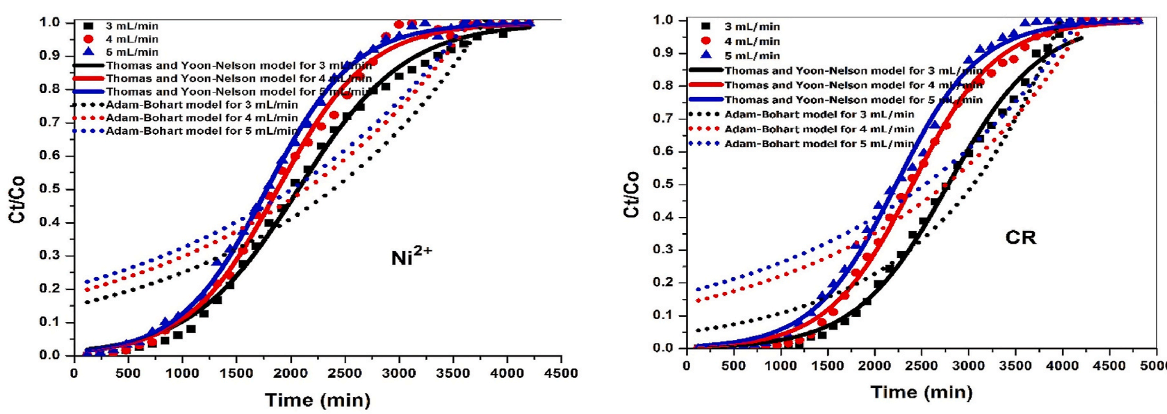
$$ln\left[\left(\frac{C_o}{C_t}\right)^{n-1} - 1\right] = \ln A - rt \quad (8)\ (\text{linear})$$




**(a)** Various initial concentrations (where flow rate = 3 mL/min, pH = 4.9, bed height = 0.20 cm)




**(b)** Various bed heights of adsorbent (where initial conc.= 25 ppm, flow rate= 3 mL/min, pH = 4.9)




**(c)** Various flow rates (where the initial conc. = 25 ppm, bed height = 0.20 cm, pH = 4.9)

**Fig. 15.** Nonlinear fitting of adsorption models, including the Yoon-Nelson, Thomas, Clark, Adam-Bohart, and Wolborska, to the experimental BTC (Breakthrough Curve) for eliminating toxicants from wastewater through a column adsorption study (Rahman et al., 2024a) and this content regenerated with the permission from Elsevier with a license number 5998600199924.

$$\text{and,}\ \frac{C_o}{C_t} = \left(\frac{1}{1 + Ae^{-rt}}\right)^{n-1} \quad \text{(9) (non-linear)}$$

Where, n is the Freundlich exponent and t, $C_0$, and $C_t$ is the time (min), adsorbate initial concentration (ppm), and final concentration (ppm) respectively. As well as A and r are parameters of kinetics equations that is obtained by plotting $\left(\frac{C_o}{C_t}\right)$ vs t slope and intercept.

### 6.4. Adams and Bohart model (ABM)

The Adams and Bohart model follow second-order kinetics and assumes that the adsorption rate is directly proportional to the adsorbent's residual capacity and the concentration of the adsorbate in a continuous fluid system, which examines the relationship between time and concentration ratio. This model is effective for initial breakthrough curve analysis rather than complete profile analysis, which assumes that surface reaction kinetics control the entire adsorption process where mass transfer should not be considered and may require more experimental data for analysis. This model is expressed by the following linear and non-linear equation (Rahman, 2024):

$$ln\left(\frac{C_t}{C_0}\right) = K_{AB}C_0t - K_{AB}N0\frac{Z}{U} \quad \text{(10) (Linear)}$$

and $\left(\frac{C_t}{C_0}\right) = \exp\left(K_{AB}C_0 t - K_{AB} N0 \frac{Z}{U}\right)$ (11) (Non-linear)

The slope and intercept can be obtained by plot $ln\left(\frac{C_t}{C_0}\right)$ vs time where $C_t$ and $C_0$ implies effluent concentration at time t (mg/L) and influent concentration (mg/L), besides $k_{AB}$ = Adams-Bohart rate constant (L/mg·min), $N_0$ = adsorption capacity per unit volume of bed (mg/L), $Z$ = bed depth (cm), $U$ = superficial velocity (cm/min) and $t$ = time (min).

### 6.5. Bed depth service time model (BDST)

The BDTS model is practical for evaluating adsorption capacity and lead time in a fixed bed column before breakthrough occurs, where the adsorbent's adsorption capacity is distributed uniformly. This model assumes that the column follows a linear isotherm, first-order kinetics and no axial dispersion under constant flow conditions, where the adsorption rate is proportional to the adsorbent's residual capacity and mass transfer is negligible. The linear and non-linear form of BDST can be represented by the following expression (Saatçi and Oulman, 1980):

$$t_B = \frac{N_0 Z}{C_0 U} - \left(\frac{1}{K_a\, C_0}\right) ln\left(\frac{C_0}{C_B} - 1\right) \quad (12)\ (\text{Linear})$$

$$\text{and } Z = \frac{C_0 U}{N_0}\left[t_{-}(B) + \frac{1}{K_a\, C_0} ln\left(\frac{C_0}{C_B} - 1\right)\right] \quad (13)\ (\text{Non-Linear})$$

where, $K_a$ = rate constant (L/mg·min), $U$ = superficial velocity (cm/min), $t_B$ = service time before breakthrough (min), $Z$ = bed depth (cm), $N_0$ = adsorption capacity per unit volume of bed (mg/L), $C_0$ = influent concentration (mg/L), and $C_B$ = breakthrough concentration (mg/L).

### 6.6. Wolborska model

The Wolborska model is effective in a fixed bed adsorption column for describing the mass transfer of solute, which is valuable for identifying the transition behavior of adsorbate in the low concentration breakthrough curve (Rahman, 2024). This is based on assumptions that define the changing concentration in a bed. The linear and non-linear form can be addressed by the following equations:

$$t = t_0 + \tau\, ln\left(\frac{C_t}{C_0}\right) \quad (14)\ (\text{Linear})$$

$$\text{and } \left(\frac{C_t}{C_0}\right) = \exp\left(\frac{t - t_0}{\tau}\right) \quad (15)\ (\text{Non-linear})$$

Where, t indicates Time (min), $t_0$ = Breakthrough time, $\tau$ = Characteristic time constant (min), $C_0$ = Initial influent concentration of the adsorbate (mg/L), and $C_t$ describes the effluent concentration at time t (mg/L).

### 6.7. Modified dose response model (MDRM)

The MDRM is an empirical model that is valid for describing the data generated from multilayer adsorption (heterogeneous adsorption system) in a fixed-bed column adsorption study. This model is effective in diminishing the error in the breakthrough curve resulting from the Thomas model. The linear and non-linear expression can be labeled by the following equation (Huang et al., 2018):

$$ln\left(\frac{C_t}{C_0 - C_t}\right) = -K\,(t - t_{50}) \quad (16)\ (\text{linear})$$

$$\text{and } \frac{C_t}{C_0} = \frac{1}{1 + e^{K(t - t_{50})}} \quad (17)\ (\text{non-linear})$$

where, K is the rate constant ($min^{-1}$), t indicates Time (min), $t_{50}$ implies 50 % breakthrough time (min), $C_0$ and $C_t$ describe the influent concentration and effluent concentration at time t (mg/L). Table 5

A non-linear curve fitting of a few mostly practiced mathematical models of fixed bed continuous column adsorption has been captured in Fig. 15. Subsequently, an effective comparison of key models of fixed bed column adsorption based on their advantages and limitations is described in Table 6. The adsorption efficiency, breakthrough time, and exhaustion time respond accordingly to decreasing or increasing process parameters. The key process parameters or several environmental factors such as effect of initial concentration of feed solution, effect of flow rate of wastewater solution through column, effect of bed height, effect of temperature, effect of pH of solution, effect of particle size of the adsorbents and their associated scientific mechanism and output/impact on the fixed bed column adsorption study are captured below in Table 7. Additionally, some significant available data, including the measure outcomes along with error metrics such as $R^2$ and $\chi^2$, after applying the considered mathematical models on the experimental BTC curve which allows users to qualitatively assess model performance and experimental fit, has been shown in Table 8 for better clarity.

## 7. Comparative study

Industrial wastewater is a significant environmental challenge because of the existence of various hazardous pollutants, including multiple dyes, heavy metals, and different toxic substances that are detrimental to human health and the environment. CNC-clay-based biopolymeric nanocomposites, particularly those based on clay and cellulose nanocrystals (CNC), are known as promising wastewater treatment materials due to their exceptional properties like biodegradability, sustainability, and high adsorption capacity. This study compares how well clay-CNC-based biopolymeric nanocomposites can be used to treat industrial wastewater. Moreover, how these materials are made and how they are effectively used to remove contaminants from wastewater. The adsorption of pollutants by clay-CNC-based nanocomposites is governed by several mechanisms, including chemisorption, physisorption, and interparticular diffusion. The hydroxyl and amino groups in the composite structure play a crucial role in binding heavy metal ions and dye molecules. For example (Rahman et al., 2023a; Majigsuren et al., 2024) studies show that Chitosan-clay composites can adsorb Cr(III) and Cr(VI) ions. This happens because these ions interact with the hydroxyl and amino groups in the composite beads. Clay-CNC nanocomposites are sometimes compared with other adsorbents, for instance zeolites and activated carbon, due to their adsorption capacity and cost-effectiveness. While activated carbon exhibits high adsorption capacities, its cost, and non-renewable nature make it less sustainable compared to clay-CNC composites. On the other hand, abundant zeolites often require complex modification processes, which can limit their practical application (Hu et al., 2021; Senila and Cadar, 2024; Srinivasan and Fogler, 1990). Despite their promising performance, several challenges remain in the large-scale application of clay-CNC nanocomposites. Further optimization of fabrication methods is needed. Future research needs to address these challenges to enhance the scalability and sustainability of clay-CNC nanocomposites used in industrial wastewater treatment (Amari et al., 2021; Ciğeroğlu et al., 2024). However, for a better understanding of the scope, contribution, novelty, significance, and selectivity of the newly proposed multifunctional FC-CNC based biopolymeric nanocomposites for wastewater treatment technology for the selective separation of hazardous toxicants from industrial wastewater bodies a comparative table is given in Tables 9 and 10. This evaluation highlights several important parameters, including adsorption capacity, regeneration cycles, adsorption rate, and effective pH range, all of which are essential for assessing the performance of these adsorbents. For instance, the cellulose nanocrystal/organic montmorillonite nanocomposite adsorbent, referred to as CNC/CTM,

**Table 5**
A comprehensive comparison between fixed bed column adsorption and batch adsorption processes based on different operational parameters while considering industrial wastewater treatment prospects (Rahman, 2024; Rahman et al., 2025b).

| S/N | Parameters | Fixed bed continuous column adsorption | Batch adsorption |
|---|---|---|---|
| 1 | Purpose | To study the response of the method in bulky industrial water treatment. | To frame the kinetics and equilibrium behavior of the adsorbent. |
| 2 | Process type | Continuous (changeable volume) | Discontinuous (fixed volume) |
| 3 | Loading of applied adsorbent | Imparts a vital role in enhancing the effectiveness of the column during operation. | Revealed no loading options during operation. |
| 4 | Important factors | Column bed height, surface contact time between the adsorbent and adsorbate, adsorbate initial concentration in the effluent, operating temperature. | Dose of adsorbent, shaking speed, adsorbate initial concentration into feed, adsorbent particle size, retention time, feed pH, temperature |
| 5 | Operative volume | Suitable for lab-scale i.e., 0.1L to 10 L | Effective in both lab-scale and industrial operations, such as 1 L to more than 10 thousand liters. |
| 6 | Adsorbate concentration | Changes along with column length | It remains constant at every point in the reactor. |
| 7 | Performance evaluation | Precisely measured through the breakthrough curve obtained from different mathematical models, a plot against experimental data. | It might be evaluated by considering the extent of adsorption capacity in equilibrium obtained from mathematical models. |
| 8 | Pressure drop | Significant | Negligible |
| 9 | Mass transfer resistance | Substantially higher | Lower (reduce film resistance due to system well mixed) |
| 10 | Temperature controlling | Demand for the installation of advanced thermal management in bulky operations. | Ease of control in a lab-scale |
| 11 | Operational flexibility | Complex | Simple |
| 12 | Maintenance issue | Required continuous monitoring to avoid clogging. | Low maintenance |
| 13 | Cost | Relatively higher due to operational complexity (sensitive device) and requires close monitoring for continuous operation. | Cost-effective for small scale operation due to process simplicity. |
| 14 | Mathematical models | Yoon–Nelson, Thomas, Clark, Wolborska, Adam-Bohart, Modified dose–response, Bed depth service time, and Yan models | Temkin, Dubinin–Radushkevich, Langmuir, and Freundlich isotherm models |
| 15 | Application frequency | Effective in bulk effluent treatment | Limited use |

exhibits a maximum adsorption capacity of approximately 69.04 mg of the herbicide diuron per gram at a temperature of 288 K. This capacity is achieved after reaching adsorption equilibrium within 318.7 min. The efficiency of CNC/CTM improves when the pH is below 6, indicating its suitability for use in slightly acidic conditions. Conversely, its adsorption efficiency decreases when the pH exceeds 8, although it remains effective across five cycles of reuse, demonstrating its potential for long-term application in environmental remediation (Ma et al., 2020). The CNC and bentonite-based nanocomposite demonstrates an adsorption capacity of 1.85 mg and maintains stability across a broad range of pH levels. The optimal adsorption efficiency for this material occurs at a neutral pH of 7, exhibiting an impressive overall adsorption efficiency of approximately 92.7 %. This high level of effectiveness highlights the potential of biopolymeric adsorbents for practical applications, especially in treating contaminated water sources (Deng et al., 2024). The economic assessment shows that CNC-clay-based biopolymeric adsorbents are cost-effective due to the availability of inexpensive raw materials, such as natural clays and CNC derived from agricultural waste, along with their proven reusability across multiple adsorption cycles (Hussain et al., 2024; Acquavia et al., 2021; Li et al., 2021). Bentonite (Bt), a 2:1 layered clay material, is recognized as a low-cost, efficient, and sustainable natural adsorbent and is priced at around $41 per ton. It is relatively inexpensive and serves as the primary component of the composite. Therefore, enhancing it with trace amounts of CNC is important for improving its effectiveness in industrial water treatment (Deng et al., 2024).

**Table 6**
A wide-ranging comparison, along with the major limitations of the models associated with fixed-bed column adsorption.

| Model | Principal parameters | Main purpose | Key assumptions | Major limitations |
|---|---|---|---|---|
| Yoo-Nelson model | $K_{YN}$, $\tau$ | Evaluate time for 50 % breakthrough | Adsorption depends on the breakthrough behavior | Can't predict adsorption capacity |
| Thomas model | $q_o$, $K_{Th}$ | Predict breakthrough behavior | Langmuir isotherm and no external or internal diffusion | Ignores resistance of mass transfer |
| Clark model | $r$, $n$ | Solved system equations | Assumes Freundlich isotherm by neglecting dispersion phenomena | Effective only for homogeneous column condition but may possible in heterogenous in certain modified conditions |
| Adams and Bohart model | $K_{AB}$, $N_0$ | Adsorbent's residual capacity | Second order kinetics and surface condition | Ignores long term adsorption behavior |
| Bed depth service time model | $K_a$, $N_0$, $Z$ | Evaluating adsorption capacity and lead time | No axial dispersion, first order kinetics | Ignores axial dispersion and fails at high concentration |
| Wolborska model | $\tau$ | Identifying the transition behavior of adsorbate in the low concentration | changing concentration in a bed | Ignore resistance of internal diffusion |
| Modified dose response model | $K$, $t_{50}$ | Describing the data generated from multilayer adsorption | Dose response behavior | Sensitive to experimental variation |

## 8. Desorption study

The desorption study of clay-CNC (cellulose nanocrystal) based biopolymeric nanocomposites is a promising area of research for industrial wastewater treatment, focusing on the elimination of various pollutant from wastewater. These nanocomposites exhibit unique properties of clay and cellulose nanocrystals (CNCs) that enhance adsorption capabilities, making them crucial for treating bulky

**Table 7**
The associated process parameters and their effects in the fixed-bed column adsorption study under varying operating conditions (Rahman, 2024; Rahman et al., 2025b).

| Process parameters | Condition | Effects |
|---|---|---|
| Flow rate (Q) | High | Breakthrough occurs earlier due to less contact time, and adsorption reaches faster, results ineffective adsorption. |
| | Low | Suitable contact time through effective utilization of the adsorbent, results in better mass transfer and enhances efficiency. |
| Bed height (H) | High | The availability of more residual adsorbent (which increases breakthrough time) and exhaustion time (which indicates higher capacity) results in longer contact time, thus showing superior efficiency. |
| | Low | Shows less surface area available for adsorption; consequently, the column saturates early. |
| Temperature (T) | High | Kinetic energy gets higher and leads to rapid saturation, where the efficiency depends on the reaction mode, whether it is exothermic or endothermic. |
| | Low | Prolonged adsorption; effective in low temperature whether it exothermic |
| pH of adsorbate | High | Enhance adsorption of anionic pollutants. |
| | Low | Enhance adsorption of cationic pollutants |
| Adsorbent particle size | large | A smaller surface area; results in slower diffusion and consequently lower efficiency. |
| | Small | A larger surface area results in better mass transfer, leading to high adsorption efficiency. |
| Initial adsorbate concentration ($C_0$) | High | Active sites were occupied early by leaving residual adsorbate. |
| | Low | Uptake of more active sites by slower loading thus leads to a higher lead time and improved efficiency. |

industrial wastewater. Some unique properties of clay and CNCs in biopolymeric matrices offer advantages over others, such as increased surface area, improved mechanical properties, and enhanced adsorption efficiency. This segment of advantages is prominent for addressing the challenges posed by industrial wastewater. A recent article by (Elahi et al., 2024) highlights the prospective of Cr-doped NiO/clay composites advancement of dye removal efficiency and CO2 capture. The composite exhibited high adsorption potential for both dye and CO2. The study also explores the nanocomposites' zeta potential and adsorption-desorption isotherm characteristics, with effective pollutant capture and sustainable desorption behavior. The findings suggest that these composites provide a reliable solution for industrial wastewater remediation. Additionally (Rahman, 2024; Sheikh et al., 2023), illustrate that Clay-CNC nanocomposites have demonstrated high efficiency in removing heavy metals, dyes, and other pollutants from industrial wastewater. For instance, composites have achieved up to 99 % removal efficiencies for certain toxicants, showcasing their potential for large-scale applications. (Rahman, 2024) This review article discusses various production methods of bionanocomposites that are based on chitosan-clay and analytical techniques. It also introduces a widely accepted and effective industrial wastewater treatment method, namely fixed-bed continuous column adsorption. Studies indicate that the performance of these nanocomposites can be affected by some factors, such as the initial concentration of contaminants, pH, and contact time (Bellaj

**Table 9**
Comparison of adsorption capacities of clay/CNC-based nanocomposites for different pollutants.

| Pollutant Type | Adsorption Capacity | References |
|---|---|---|
| Pb(II) | 351.04 mg/g | Shen et al. (2022) |
| Cr(III) | 12.4 mg/g | (Elahi et al., 2024a) |
| Cr(VI) | 17.31 mg/g | (Majigsuren et al., 2024b) |
| Methylene Blue | 50.0 mg/g | (Bellaj et al., 2024; Ibrahim et al., 2024) |
| Congo Red | 478.3 mg/g | Rahman et al. (2024a) |
| $Ni^{2+}$ | 101.24 mg/g | Rahman et al. (2023a) |
| $Cu^{2+}$ | 94.14 mg/g | Rahman et al. (2023a) |
| $Ni^{2+}$ | 328.7 mg/g | Rahman et al. (2024a) |
| $Ni^{2+}$ and Eosin Y dye | 186.42 & 238.37 mg/g | Rahman et al. (2024f) |
| $Pb^{2+}$ and CR dye | 538.91 & 455.70 mg/g | Rahman et al. (2024d) |

**Table 8**
A list of significant parameters have been obtained by applying the Thomas model, the Yoon–Nelson model, the Clark model, the Bohart–Adams model, and the Wolborska model at various operating conditions regarding $Ni^{2+}$ ions by following nonlinear deterioration analysis. This table and data has been regenerated with permission from Rahman et al., 2024g.

| Operating Condition | Q (mL/min) | 3 | 3 | 3 | 2 | 3 | 4 | 2 | 2 | 2 |
|---|---|---|---|---|---|---|---|---|---|---|
| | **Ci (ppm)** | 30 | 40 | 50 | 40 | 40 | 40 | 30 | 30 | 30 |
| | **Bed height (cm)** | 1.0 | 1.0 | 1.0 | 0.5 | 0.5 | 0.5 | 0.5 | 1.0 | 1.5 |
| **Thomas model** | **$q_0$ (mg/g)** | 120.98 | 169.18 | 201.91 | 108.23 | 149.36 | 213.83 | 238.16 | 193.26 | 113.21 |
| | **$K_{th}$** | 0.0107 | 0.0126 | 0.0150 | 0.0135 | 0.0130 | 0.0111 | 0.0129 | 0.0903 | 0.0108 |
| | **$R^2$** | 0.997 | 0.991 | 0.979 | 0.994 | 0.981 | 0.980 | 0.994 | 0.994 | 0.997 |
| | **$\chi^2$** | 0.0003 | 0.0010 | 0.0025 | 0.0007 | 0.0022 | 0.0026 | 0.0008 | 0.0008 | 0.0004 |
| **Yoon–Nelson model** | **$\tau_{50}$ % (min)** | 1459.75 | 1443.23 | 1434.66 | 1531.61 | 1473.05 | 1389.39 | 1296.23 | 1365.14 | 1442.18 |
| | **$K_{YN}$** | 0.0033 | 0.0034 | 0.0031 | 0.0049 | 0.0040 | 0.0038 | 0.0041 | 0.0040 | 0.0039 |
| | **$R^2$** | 0.997 | 0.991 | 0.979 | 0.994 | 0.982 | 0.980 | 0.994 | 0.994 | 0.997 |
| | **$\chi^2$** | 0.0003 | 0.0009 | 0.0024 | 0.0007 | 0.0022 | 0.0025 | 0.0008 | 0.0008 | 0.0004 |
| **Adam–Bohart model** | **$K_{AB}$** | 0.0032 | 0.0034 | 0.0032 | 0.0043 | 0.0038 | 0.0033 | 0.0031 | 0.0032 | 0.0034 |
| | **$N_0$** | 3035.72 | 3122.91 | 3398.34 | 3181.21 | 3308.01 | 3745.92 | 52912.09 | 4419.09 | 3998.36 |
| | **$R^2$** | 0.949 | 0.964 | 0.968 | 0.961 | 0.964 | 0.951 | 0.937 | 0.943 | 0.946 |
| | **$\chi^2$** | 0.0057 | 0.0041 | 0.0037 | 0.0047 | 0.0042 | 0.0061 | 0.0082 | 0.0073 | 0.0066 |
| **Wolborska model** | **β** | 0.0033 | 0.0034 | 0.0032 | 0.0043 | 0.0038 | 0.0033 | 0.0031 | 0.0032 | 0.0034 |
| | **$N_0$** | 74.70 | 99.88 | 124.80 | 92.10 | 93.86 | 98.82 | 116.96 | 98.98 | 84.50 |
| | **$R^2$** | 0.949 | 0.964 | 0.968 | 0.961 | 0.964 | 0.951 | 0.937 | 0.943 | 0.946 |
| | **$\chi^2$** | 0.0057 | 0.0041 | 0.0037 | 0.0047 | 0.0042 | 0.0061 | 0.0082 | 0.0073 | 0.0066 |
| **Clark model** | **A** | 923.29 | 1527.98 | 2235.89 | 1165.09 | 1890.34 | 2789.16 | 3428.97 | 2169.04 | 2364.09 |
| | **r** | 0.00413 | 0.00569 | 0.00961 | 0.00784 | 0.01243 | 0.01252 | 0.0065 | 0.0056 | 0.0045 |
| | **$R^2$** | 0.999 | 0.997 | 0.996 | 0.998 | 0.996 | 0.989 | 0.998 | 0.996 | 0.997 |
| | **$\chi^2$** | 0.0001 | 0.0004 | 0.0011 | 0.0002 | 0.0006 | 0.0014 | 0.0003 | 0.0004 | 0.0003 |

**Table 10**
A comprehensive table of the key data on clay- CNC-based adsorbents, including material, adsorbate, $q_{max}$ (mg/g), effective pH, adsorption time (min), regeneration cycles, and efficiency (%).

| Filter materials/adsorbents | Adsorbate | $q_{max}$ (mg/g) | Effective pH Range | Adsorption Rate (min) | Regeneration Cycles | Efficiency (%) | Reference |
|---|---|---|---|---|---|---|---|
| CNC/CTM Nanocomposite | Diuron | 69.04 | increase at pH < 6, decrease at pH > 8 | 318.7 | ≥5 | 82.32 % | Ma et al. (2020) |
| CNC@Bentonite | Cr(VI) | 1.85 | Wide pH range with optimum results at pH 7. | Significant adsorption was observed within 2 min and complete equilibrium at 30 min. | 4 | 92.7 % | Deng et al. (2024) |
| PVA/Chitin/NCC (PVA/CT10/NCC/MA30) | Methylene Blue | 467.5 | Increase from pH 3 to 8 and maximum adsorption at pH 9 | 360 | 5 | 83.67 ± 1.08 % | Mok et al. (2020) |
| ZIF-8/CNC nanohybrid | Cd(II) | 423 | 7 | Rapid initial uptake in first 15 min, equilibrium reached within 120 min | 5 | 93.8 % | Mohammadi et al. (2024) |
| CNC-Pectin Composite | Uranium (VI) | 100 | Wide pH range(4−10) | Equilibrium is achieved within 2 min | 4 | >98 % | Nayak et al., |
| Bentonite-Clay/CNT | Textile Dyes | 550 | 3 | 120 | _ | 89.9 | Jamil et al. (2023) |
| Poly(acrylic acid)-modified poly (glycidylmeth-acrylate)-grafted nanocellulose (PAPGNC) hydrogel | Trypsin | 140.65 | 6.5 | 90 | 4 | 87 % | Rout et al. (2021) |
| Cr-doped NiO/Clay Biocomposite | RY-18 dye | 398 | 8 | Measured after ~120 | 3 | 91.28 % | Elahi et al. (2024) |
| clay-CNT nanocomposite (CCN) | Lead | 9.43 | 6 | 120 | _ | 88–90 % | Yadav et al. (2019) |

et al., 2024). Under the best optimal conditions of a 20 mg dose, at pH 2, a reaction duration of 30 min, and a dye concentration of 20 mg/L, approximately 92 % of the dye removal was achieved. The adsorption of dye by the composite followed the Langmuir isotherm model, while the process of dye removal was governed by a pseudo-second-order model. Thermodynamic data for the adsorption indicated that the enthalpy change ($\Delta H$) was +8.82 kJ/mol, and the Gibbs free energy change ($\Delta G$) was less than zero. This indicates that the dye adsorption process was both spontaneous and endothermic. Meanwhile, clay-CNC-based biopolymeric nanocomposites offer significant advantages in wastewater treatment. But there are also some challenges remaining in large-scale implementation, such as the regeneration and reuse and long-term stability and environmental impact of the composite. Additionally, using these nanocomposites in wastewater treatment systems requires careful consideration of cost-effectiveness and operational feasibility. Future research should optimize the synthesis processes and explore new composite formulations to enhance performance and sustainability in industrial applications. A substantial debt regarding such confines as well as their remediation techniques for cutting-edge research for the future era has been proposed in this review. However, in a nutshell, for a better understanding of the scope, contribution, significance, relevancy, and novelty of the regeneration/desorption study of the considered CNC-based biopolymeric nanosorbents regarding the real-time bulky industrial wastewater treatment, an example is shown in Fig. 16 and Table 11.

## 9. Conclusion

Production of novel and ecofriendly multifunctional biopolymeric nanoadsorbents from the low cost natural resources such as lignocellulosic agrowaste biomass, natural clay, etc., by applying economical and sustainable technology for the effective treatment of hazardous industrial wastewater is a new trand and currently fortified the researchers globally to mitigate the environmental pollution. Therefor, in this current study, the natural plant based agrowaste biomass derived CNCs and naturally occurring white clay based polyfunctioal FC-CNC bionanoadsorbents production routes have been proposed additionally their characterization, and applications modes for realtime bulky industrial wastewater treatment have also been discussed comprehensively. Because the waste biomass derived FC-CNC based multifunctional bionanoadsorbents have confirmed more greater adsorptive removal performance/efficiency regarding the selective separation of the hazardous toxicants from the bulky wastewater bodies during the real-time experimental sessions. It is important to mention that the multifunctional FC-CNC bionanoadsorbents would be an auspicious candidate for the replacement of the fossil-based nonbiodegradable synthetic adsorbents/composite materials for wastewater treatment and to reduce the environment pollution because of their cost effectiveness, availability, greater performances, ecofriendly nature, higher surface area, and outstanding physicochemical, thermomechanical and morphological properties. This study is devoted to develop and provide report on the noteworthy modification techniques of the selected precursors like cellulose and natural clay, several state-of-the-art production techniques of the FC-CNC bionanoadsorbents, their categorization approaches, in addition to their mode of application addressing the bulky industrial wastewater treatment during the experimental session for better clarity. The decisive consequences that earlier have been offered in this article would be looking abundant to produce a new, innovative, and beneficial class of environmentally friendly biobased polymeric nanoadsorbents for wastewater purification for the development of a greener environment as well as cleaner society. Conversely, it has been predicted that if the previously modified CNCs could be effectively integrated on the functionalized natural clay while fabricating superactive polyfunctional bionanoadsorbents then the fabricated FC-CNC bionanoadsorbents must provide an extraordinary adsorption performance addressing the selective separation of the hazardous toxicants simultaneously from wastewater bodies due to synergistic adsorption mechanism which indicates all the chemisorption, monolayer and multi-layer physisorption along with deep penetration. Notably, the produced FC-CNC bionanoadsorbents have possessed some limitations concerning the real-time applications in the purifications of the bulky industrial wastewater through the continuous mode of adsorption process. Such as: the insufficiency of the appropriate regeneration tactics which could cope with the use of the reprocessed biopolymeric nanoadsorbents without impeding their unique microstructure, overall properties, potential and adsorption proficiency. Moreover, by offering a robust database this critical review could enlarges the instinct to attain numerous convenient FC-CNC

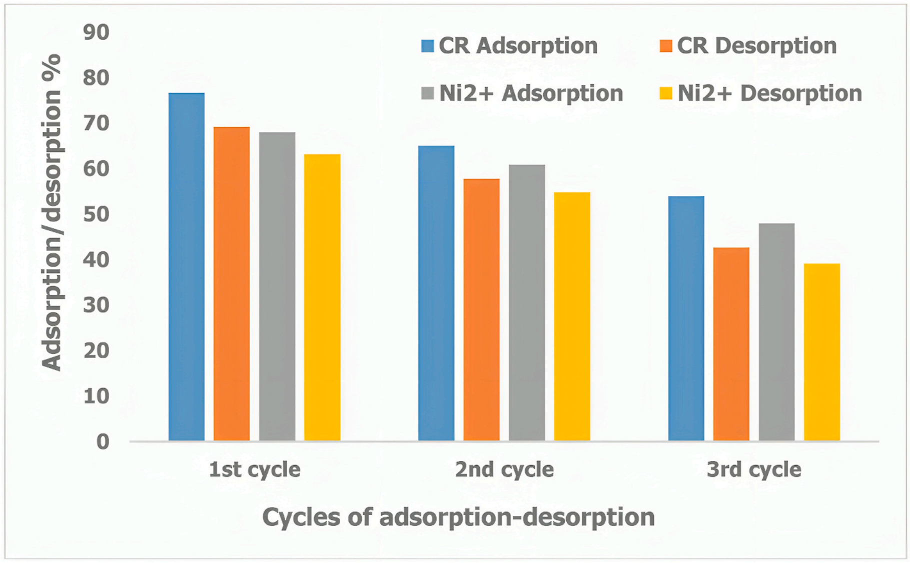



**Fig. 16.** The newly created FC-CNC biopolymeric nanosorbents adsorption-desorption cycles following repeated regeneration and application concerning the targeted toxicants, such as $Ni^{2+}$ and CR dye during the experimental session of (Rahman et al., 2024a) regenerated with the permission from Elsevier with a license number 5998600199924.

bionanoadsorbents by reducing the reliable inadequacies addressing the real-time crude industrial wastewater refinement process.

## 10. Challenges and future prospective of research

Industrial wastewater has been biggest concern in recent years. This has very meaningful impact in the water footprint as well as eco-system of the environment. Industrial wastewater contains different toxicant. Clay and CNCs can be functionalized to greatly improve their surface qualities, which will boost their capacity to adsorb a wide range of contaminants, such as organic pollutants, dyes, and heavy metals, nanoplastics, pesticides, that are common in industrial effluents. Especially presence of heavy metal heavy metals in water can have significant adverse effects on both human health and the environment. Also rapid contamination by waste water to the ground water can create public health concern. Combining clays with cellulose nanocrystals to enhance properties such as mechanical strength, barrier performance, and thermal stability. This type of clay CNC based nanocomposite can be derived from wastage like bamboo, cotton, algae, wood, jute, okra stick, mushroom, sugarcane bagasse etc. that are eco-friendly and naturally available also. Reusing waste biomass to CNC support economical sustainability as a result dependency on renewable energy reduces. Functionalized clay-based CNC bio-polymeric nanocomposites seem promising; they could transform industrial wastewater treatment by offering economical, effective, and sustainable solutions that tackle environmental and regulatory issues. But in practical industrial waste water contains different hazardous toxicants with individual properties based on pH, concentration etc. While many studies focusing on single pollutants. Considering these issue carefully bio-polymeric nanocomposite is studying. Clay-CNC nanocomposites with other treatment technologies like membrane filtration or biological process could result in beneficial effects that raise the effectiveness of treatment as a whole. Beside industrial effluent varies regarding waste type, concentration of the waste water, temperature all have to consider before fabrication of adsorbent. Furthermore, because these materials are biodegradable and derived from renewable feedstocks, they lessen the environmental impact of conventional wastewater treatment systems. This is in line with the increased emphasis on sustainability. Additionally, it is also noteworthy that nowadays researchers from all over the world have trying to develop their research for zero waste. Hence, production of multifunctional FC-CNC based biopolymeric nanoadsorbents from the agricultural waste biomass would be very much new, innovative, and beneficial ones to mitigate the environmental pollution and cost effectiveness issue simultaniously by reducing the solid waste, and water pollution. Furthermore, the used adsorbents would be disposed by land filling or by developing building/construction materials, or by creating supercapasitor or something like that for better yield and greater ecogeological impact in future research.

## CRediT authorship contribution statement

**Md. Mahmudur Rahman:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Songita Rani Adhikary:** Writing – original draft, Software. **M Mohinur Rahman Rabby:** Writing – original draft, Methodology. **Md. Mahafujul Hassan:** Writing – original draft, Data curation. **Salah Knani:** Funding acquisition. **Syed Hasibul Akhfer:** Writing – original draft, Resources. **Md. Khalid Al Zuhanee:** Writing – original draft, Methodology, Data curation.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## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

**Table 11**
Summary of the different regeneration/desorption processes according to the pollutant adsorbed.

| Adsorbent | Contaminant | Reagent | Cycles | Efficiency | Reference |
|---|---|---|---|---|---|
| TOCNF/chitin | MB | 0.05 HCl | 5 | 99 % | Zhang et al. (2019) |
| CNEF/APS | $Ni^{2+}$, $Cu^{2+}$, $Cd^{2+}$ | 0.1 M NaOH | 2 | 155 % - $Ni^{2+}$ 111 % -$Cu^{2+}$ 101 % - $Cd^{2+}$ | Hokkanen et al. (2014) |
| CNC-alginate | MB | 1 M HCl | 5 | – | Mohammed et al. (2015) |
| CNF-MAA-MA | $Cd^{2+}$, $Pb^{2+}$, $Zn^{2+}$, $Ni^{2+}$ | EDTA | 2 | 98 % | Long et al. (2018) |
| CNF | $Zn^{2+}$, $Ni^{2+}$, $Co^{2+}$, $Cd^{2+}$ | 1 M $HNO_3$ | 2 | 95.3 % - $Zn^{2+}$ 95.8 % - $Ni^{2+}$ 103.6 % - $Co^{2+}$ 95.5 % - $Cd^{2+}$ | Hokkanen et al. (2013) |
| CMC/CS-SGO | Sulfonamides | $HNO_3$ | 5 | 87 % | Liu et al. (2021) |
| CS-MC | Crystal violet $Pb^{2+}$ | 0.2 M NaOH | 5 | 78.09 % - CV 72.21 % - $Pb^{2+}$ | Rahman et al. (2023d) |
| Silylated CNF | Dodecane | Toluene | – | ~100 % | Zhang et al. (2014) |
| Thiol modifie CNF | $Cr^{6+}$, $Pb^{2+}$ | 0.05 M EDTA | 3 | 93 % - $Cr^{6+}$ 95 % - $Pb^{2+}$ | Yang et al. (2014) |
| CNC-MC | $Ni^{2+}$ CR dye | 0.2 M NaOH | 3 | 63.09 % - $Ni^{2+}$ 69.13 % - CR | Rahman et al. (2024a) |
| CNF | $Cd^{2+}$, $Pb^{2+}$, $Ni^{2+}$ | 0.5 M $HNO_3$ | 4 | 99.2 % - $Cd^{2+}$ 96.8 % - $Pb^{2+}$ 90.6 % - $Ni^{2+}$ | Kardam et al. (2014) |

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## Data availability

Data will be made available on request.

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