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Nanocomposites of Magnetically Retrievable Cellulose and Metal Oxide Frameworks for Efficiency in Concurrent Pollutant Removal

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Abstract

The complexity of industrial wastes has increased significantly with the increase in number of pollutants that are discharged in a single waste stream, and conventional single pollutant treatment processes have become largely ineffective. This scientific work offers a comprehensive study of the synthesis, characterization and application of magnetically recoverable metal-organic frameworks (MOF) and transition metal oxide (TMO) composites based on cellulose. The intrinsic biocompatibility, hydrophilicity and functional group density of nanocellulose makes these composite materials more suitable than unmodified metal-organic frameworks to overcome their structural vulnerability and tendency for agglomeration. The superparamagnetic iron oxide nanoparticles (SPIONs), primarily magnetite Fe_3O_4 particles, overcome the technical challenge of post-treatment separation of the solid adsorbents from the liquid solution by being easily recovered from the liquid by external magnetic field. The composites of MCNC presented significant specific surface area (Type IV isotherms and mesoporous structure) that leads to outstanding adsorption property. These materials have shown excellent performance for the simultaneous removal of heavy metal cations (e.g., Pb(II), Cu(II), As(III)), synthetic dyes (e.g., methylene blue, malachite green) and pharmaceutical micropollutants (e.g., tetracycline). The adsorption phenomenon in these complex multisolute systems is mainly described by pseudo-second order kinetic model and follows the Langmuir and extended Langmuir isotherm models indicating that the primary removal phenomenon is monolayer chemisorption. The synergy between electrostatic attraction, Lewis acid-base interactions, π-π stacking and hydrogen bonding allows distinct contaminants to bind to different active sites without the complete competitive exclusion of other contaminants, mechanistically. Moreover, thermodynamic evaluations validate the spontaneity of the adsorption mechanism. Due to their outstanding chemical stability and reusability, these magnetic composites are a highly promising, scalable and sustainable approach for comprehensive purification of complex wastewater systems.

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1. Introduction: Pollution of a variety of industrial and agricultural wastes into fresh water has led to a major environmental challenge in the 21st century. Today's wastewater is not made up of

single pollutants – it is a complex mix of multiple components that contain all sorts of heavy metals, synthetic organic dyes, and persistent pharmaceuticals, such as antibiotics. Co-occurrence of such contaminants creates greater environmental risks. Heavy metals are very dangerous, persistent in the environment and have the potential to bioaccumulate, while organic dyes and antibiotics disrupt the photosynthesis in the water and increase the rate of antimicrobial resistance. Therefore, the development of resilient remediation systems that are able to effectively and simultaneously remove these vastly different contaminants is essential for environmental engineering and public health.

Of all the physicochemical treatment methods (membrane filtration, chemical precipitation, advanced oxidation and electrodialysis), adsorption is known for its simplicity, low cost and highly effective performance at low concentrations. The structural properties and surface chemistry of the adsorbent material, and the binding strength, are the key factors in determining the effectiveness of any adsorption system. While traditional adsorbents, such as activated carbon, unaltered biochar, and pure metal oxides, can exhibit limited specific surface areas, limited functional adaptability, and non-selective adsorption characteristics, particularly when the environment includes multiple solutes, where the competition for adsorption can greatly reduce overall effectiveness.

Recognizing the limitations of traditional materials, metal-organic frameworks (MOFs) have emerged as a novel class of highly crystalline, porous coordination polymers. MOFs consist of inorganic metal nodes connected together by multidentate organic connectors with a broad spectrum of functional groups, a remarkably high number of which can be modified, and a very high specific surface area, in addition to a highly tunable pore structure. Despite their high potential for selective molecular capture, pristine MOFs can often lack structural fragility, hydrolytic durability, and strong tendency to aggregate into non-porous macro-clusters when placed in aqueous solutions to be effectively used in large-scale wastewater treatment.The combination of MOFs with environmentally friendly, organic-based biopolymers, especially nanocelluloses, has resulted in a novel structural solution that addresses these shortcomings. Cellulose is the most abundant natural polymer on the earth, offering a sustainable, structurally strong and highly renewable framework which is environmentally friendly. The hydroxyl groups in the cellulose framework are well suited for in-situ growth of MOF crystals by providing optimal anchoring and nucleation sites, while, after some modification, there are also carboxyl functional groups. This interaction hinders MOF clustering, enhances access to active sites, and significantly enhances the hydrolytic stability of the resulting composite.

One of the major problems hampering the use of nanoscale adsorbents is the separation of solids and liquids post-treatment. Conventional filtering or centrifugation processes are extremely energy-intensive, time-consuming and prone to additional environmental pollution when attempted to extract the MOF particles from large amounts of processed effluent, which are depleted by just a few nanometres. If the few nanometres-scale MOF particles are to be extracted from the large volume of processed effluent, conventional filtering or centrifugation processes would be extremely energy intensive, time consuming and highly polluting of the environment. The use of magnetic metal oxide nanoparticles (MNPs), specifically magnetite (Fe_3O_4) within the framework of cellulose-MOF offers a revolutionary advantage for operation. The resulting magnetic cellulose nanocrystal (MCNC) composites allow for fast, energy-saving and complete recovery of the loaded adsorbent by using an external magnetic field easily.

The purpose of this paper is to give a thorough survey on the synthesis, characterization of the structures and the wide variety of applications of MCNC@MOF and metal oxide nanocomposites to the environment. This work provides a thorough study of the kinetic, thermodynamic, and mechanistic characterization of the simultaneous removal of pollutants,

highlighting the potential of the combination of nanocellulose, MOFs, and magnetic nanoparticles as an outstanding water remediation solution in a sustainable and circular economy framework.

2. Review of Literature: Through the evolution of highly developed adsorbent material for wastewater treatment, one can see a steady progression towards higher surface areas, higher densities of functional groups, and better recoverability. It is well documented in the literature that natural cellulose and derivatives were the first natural materials that were used as the main bio-adsorbents. It was early recognized that cellulose microfibrils (CMF) and cellulose nanocrystals (CNC) are compatible with biological systems, easily chemically modified, and water affinity. However, the cellulose substances without any defect generally showed only slight adsorption properties and these were considerably influenced by the presence of inherent hydroxyl groups. In order to improve efficacy, investigation focused on the chemical modification, which led to the development of improved retention for some heavy metal cations or anionic dyes with electrostatic interactions by carboxymethylation, sulfonation and amine grafting.

At the same time, there were developments in cellulose chemistry that effectively revolutionized the study of adsorption, namely the development of metal-organic frameworks. Several types of metal-organic frameworks (MOFs), including UiO-66 (based on Zr), HKUST-1 (Cu) and ZIF-8 (Zn), have been shown to have remarkable sequestration potential in pioneering research. The literature shows that pure MOFs have outstanding adsorption capacity, which is sometimes higher than 1000 mg of adsorbate per gram of adsorbent, due to carefully designed pore geometry to promote molecular sieving and targeted functional group interactions. MOFs with sulfone based soft donors showed a remarkable attraction to soft acids like mercury and the removal efficiency was 99.2% in the absence of lighter competing ions. However, the literature also reported the critical operational limitations of pure MOFs in continuous-flow aqueous environment due to their powdery nature, poor mechanical robustness, and high separation challenges.

The development of CelloMOFs (cellulose-metal-organic framework composites) has emerged as a game-changing breakthrough designed to overcome these structural limitations. The study emphasizes the important finding that the specific surface area and structural porosity of the cellulose is significantly improved when MOFs are incorporated onto it, as well as that the MOF is structurally stabilized. A significant investigation found that the surface area of Cu-BTC was greatly enhanced from 78.4 m^2/g to 965.8 m^2/g and the adsorption capacity was also improved when the Cu-BTC was combined with a cellulose acetate membrane. Furthermore, the introduction of cellulose microfibrils has shown to enable consistently dispersed MOF crystals at the nanoscale and thus prevent against the common blockages of the microporous openings in pristine MOF powders.

The body of research on the transition to systems that are magnetically retrievable is the most recent, and most significant, in this area. To achieve precise separation, on-demand, the superparamagnetic Fe_3O_4 nanoparticles have been introduced in the cellulose matrix before the MOF functionalization. Hasan et al. demonstrated that cellulose nanocrystals derived from wood pulp are excellent templates for in-situ co-precipitation of iron oxides and the obtained magnetic substrates are very stable without nanoparticle aggregation, owing to the sterically stabilizing effect of cellulose nanocrystals. The latter functionalization with MOFs has resulted in ternary systems (Cellulose/ Fe_3O_4/MOF) with remarkable environmental efficacy.

Recent research has increasingly focused on the effects of concurrent adsorption of multisolute systems, which more closely mimic real wastewater compositions. Research shows that appropriately optimized magnetic MOF composites can be used to effectively and simultaneously remove several different contaminants. For instance, a nanocomposite based on UiO-66 and ZIF-8

is carefully designed to capture complex mixtures of heavy metals (e.g. Cr(VI) and Pb(II)) and to be resistant to antibiotics (e.g. tetracycline) and colourants. According to the literature, the bifunctional composites take advantage of different binding sites on the particles for different shapes of molecules, and are representative of the cutting edge of modern environmental materials research.

3. Materials and Methods: The comprehensive evaluation of the MCNC@MOF and related metal oxide nanocomposites for the simultaneous removal of pollutants requires a strict scientific approach and many aspects. This involves careful chemical synthesis of the nanocomposites, attention to detail with the physicochemical characterization, and systematic batch adsorption experiments coupled with advanced mathematical modelling.

Mostly sequential in-situ templated growth methods are used in the production of these ternary composites. The first step is the preparation of magnetic cellulose nanocrystal (MCNC) core. In general, longer ultrasonication is used to homogeneously disperse cellulose nanocrystals or micro-fibrils in a solution of deionized water. In-situ co-precipitation of stoichiometric amounts of salts of Fe(II) and Fe(III) (such as $FeCl_2$ and $FeCl_3$) is the most common method of magnetic functionalization, which is carefully done in an inert nitrogen/argon environment to prevent the oxidation of the ferrous ions. By slowly adding an alkaline precipitating agent (e.g., NH_4OH or NaOH) to the mixture under strong mechanical stirring and high temperatures (usually 80-90 °C), magnetite (Fe_3O_4) nanoparticles can be directly formed on the abundant hydroxyl and carboxyl functional groups of the cellulose backbone.

The metal-organic framework structure is formed in-situ on the stability of MCNC matrix, which completes the composite architecture. The purified MCNC is added to a specific solvent (e.g., DMF, MeOH, or H2O) and with the required amount of natural metal precursors such as Zn(NO3)2·6H2O, ZrCl4, or other organic connectors like benzene-1,3,5-tricarboxylic acid, 2-methylimidazole, or terephthalic acid to synthesize the desired MOF species, such as Zn-BTC, UiO-66, or ZIF-8. The functional groups on the magnetic cellulose act as accurate structure-directing anchor sites for the use of solvothermal methods in Teflon-lined autoclaves or just long mechanical stirring at room temperature. These functional sites are closely related to the metal nodes, which promote the crystal growth of MOF in a large, dense and uniform manner in the magnetic cellulose framework to wrap the magnetic core with a highly porous shell.

Physicochemical characterisation is essentially important to ensure the integrity of the structural morphology, surface chemistry of the fabricated materials. To confirm the successful incorporation of functional groups, Fourier Transform Infrared Spectroscopy (FTIR) is used, which shows the characteristic absorption peaks of the cellulose framework, the strong Fe-O stretching vibrations of the magnetite core (~570 cm^{-1}), and the distinctive carboxylate or imidazole coordination bonds characteristic of the MOFs. Powder X-ray Diffraction (PXRD) is used to check the purity of the crystalline phase which shows the appearance of the cubic spinel structure of the inorganic Fe_3O_4 nanoparticles and the well-ordered topological framework of the MOF without any phase interference.

Morphological integration and topological examination is done using Scanning and Transmission Electron Microscopy (SEM/TEM) along with Energy Dispersive X-ray (EDX) spectroscopy. These methods demonstrate the nanoscale distribution and confirm the uniform distribution of metals (Fe, Zn, Cu, Zr) on the cellulose microfibrils and that there is no significant agglomeration of the MOF crystals. The Brunauer-Emmett-Teller (BET) surface area assessment and Barrett-Joyner-Halenda (BJH) pore size distributions are essential to measure the significant increase in specific surface area, overall pore volume as well as the presence of micro- and

mesoporosity as a result of incorporation of MOFs. Finally, the experiment is carried out at room temperature to determine the saturation magnetization (M_s) of the composite, which is necessary to ensure the sensitivity to external magnetic field for quick and energy-efficient retrieval operation.

Meticulous controlled batch studies are used to evaluate the adsorption effectiveness of the nanocomposites. A certain amount of the adsorbent MCNC@MOF is introduced into synthetic wastewater containing a predetermined initial concentration of heavy metal (such as Pb^{2+}, Cu^{2+}), an anionic or cationic dye (like methyl orange, methylene blue), or pharmaceutical agent (such as tetracycline). Methodological variations such as initial pollutant concentration and operation temperature, contact time, and initial solution pH are carefully varied to identify their respective roles on adsorption capacity.

In particular, for simultaneous removal investigations, multisolute systems are prepared, such as the binary system of heavy metal and antibiotic, and the ternary system of different dyes, which simulate the complex industrial wastewater. The concentrations of the target adsorbates in the remaining unadsorbed concentrations are regularly analysed by UV-Vis spectrophotometry (for organic dye and antibiotics) or by Atomic Absorption Spectroscopy (AAS) or Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (for heavy metal ions).

4. Discussion:

The superb efficiency of the magnetically retrievable cellulose and metal-organic framework nanocomposites in the simultaneous removal of complicated contaminants is related to the meticulous coordinating and multi-tiered synergy among their constituent components. The elucidation of the precise chemical interaction mechanisms, structural advantages and thermodynamic considerations provides a crucial perspective on their superiority to conventional adsorbents.

4.1. Mechanisms of Pollutant Interactions in Concurrent Systems: The large number of binding sites engineered within the MCNC@MOF structure allows for the simultaneous adsorption of a wide range of pollutants such as small highly charged metal cations, large and complex antibiotic molecules and large steric dye molecules. The pH of the aqueous medium is a very important factor in the primary removal process, as it affects the chemical form of the contaminants to be removed from the liquid phase and the surface charge of the solid adsorbent.

Strong electrostatic forces and coordinate Lewis acid-base interactions mainly contribute to the extraction of heavy metals like Pb^{2+}, Cd^{2+} and Cu^{2+}. The disorganized carboxyl (-COOH), hydroxyl (-OH), and purposefully introduced amino (NH_2) groups on the cellulosic backbone and the organic linkers in the MOF can act as good electron donors with strong affinity to electron-poor and positively charged metal ions. Deprotonation of these functional groups when the pH of the solution is approaching neutrality causes the overall negative surface charge of the composite to increase significantly and hence the electrostatic attraction to metallic cations is increased significantly. However, in the case of Cu^{2+}, increasing the pH to very high pH (e.g., > 6-7) causes the metal ion to be removed primarily by bulk chemical precipitation, rather than by adsorption on the actual surface, because of the formation of complex hydroxide species (e.g., $Cu(OH)_2$ or $Cu(OH)_3^-$).

However, more complex organic compounds such as the tetracycline antibiotics and artificial colors (e.g., methylene blue, malachite green) form non-covalent interactions with the nanocomposites over a broader range. Some of the MOF frameworks have large and tunable cavities, such as UiO-66 or ZIF-8, where antibiotics can be sterically tailored to be physically "pore-filled" or be confined in the cavity. In the MOFs, the aromatic rings of the organic linkers (e.g., benzene-1,3,5-tricarboxylic acid or 2-methylimidazole) undergo extensive and very stable π-π stacking interactions with the aromatic rings of the dyes and tetracycline compounds. Concurrent

hydrogen bonding between the electronegative heteroatoms (N, O) found in the contaminants and the many hydrogen-rich functional sites on the cellulose/MOF interface enhances the adhesion.

This is simultaneous sequestration in a multisolute mixture and the processes are not competitive. A heavy metal ion is able to interact directly with a localized metal site in the MOF, or a specific carboxylic acid in the cellulose, while a large tetracycline molecule is simultaneously bound into an adjacent mesoporous cavity by π-π stacking and hydrophobic interactions. This structural phenomenon provides the understanding of the good agreement between the experimental competitive adsorption data and the extended Langmuir models: the competing adsorbates are usually not competing for the same active sites, highlighting the bifunctionality of the composite structure.

4.2. The Synergistic Role of Nanocellulose and Magnetite: Despite their extremely high specific surface area and custom-designed active binding sites the MOFs are too fragile to be used in high-shear, large volume water systems under continuous agitation. The MOF crystals are incorporated into nanocellulose, creating a highly flexible and stable structure that is able to effectively withstand mechanical fracture and hydrolytic degradation of the crystals. Furthermore, the cellulose microfibrils make an excellent dispersion matrix. In isolation, magnetic Fe_3O_4 nanoparticles have very high surface energy and readily coalesce into large, dense macro-clusters when subjected to strong dipole-dipole interactions, thus effectively reducing the surface area of the nanoparticles. The close proximity of functional groups on the cellulose matrix spatially hinders this clustering, ensuring that the magnetic nanoparticles are maintained at the nanoscale even after assembly, with ideal magnetic reactivity and catalytic accessibility.

The magnetic recoverability of the Fe_3O_4 core marks a game-changer in terms of operation and cost. Traditionally, the separation of used nano-scale adsorbent granules after they have been depleted from immense amounts of processed effluent is an energy-consuming process that requires expensive microfiltration systems. These composites can quickly be removed from the treated water, and in this way, sludge formation is avoided, expensive filtration systems can be avoided, and the water can be quickly transported to regeneration facilities.

4.3. Stability, Regeneration, and Economic Viability: However, an advanced adsorbent can only be transferred from the theoretical level to its practical application if it has a large initial adsorption capacity and a high regeneration durability. Literature clearly shows the exceptional chemical and structural stability of MCNC@MOF nanocomposites. Contaminated composites containing hazardous metals and organics can be effectively desorbed using fairly mild and inexpensive eluents. For example, saturated composites can be rinsed with dilute hydrochloric acid (e.g., 0.2 M HCl) or methanol, thereby effectively disrupting the specific metal-ligand coordinate bonds or electrostatic interactions, which in turn expels the heavy metals and organics in the saturated composites into a concentrated, manageable waste stream.Importantly, several research results showed that these nanocomposites retained their original maximum adsorption capacity by 80–94% after 5–7 consecutive cycles of full saturation, magnetic retrieval and chemical desorption. This slight decrease in capacity observed across cycles is attributed primarily to non-reversible incorporation of persistent organics in the deep, sterically restricted microporous space, or to progressive and local degradation of the MOF organics linkers, under very acidic stripping conditions. It is worth noting, however, that the considerable cyclic reusability effectively compensates any initial synthetic cost for the transition metal precursors and functionalized cellulose, which makes the MCNC@MOF structure economically attractive for large-scale municipal or industrial continuous-flow operations.

Conclusions and Suggestions: Continuous development and growing complexity of industrial pollution require remediation systems that are robust in their capturing ability, flexible in their usage, and highly durable in their operation. This detailed study has shown that the rationally designed fabrication of the magnetically recoverable nanocellulose (MCNC) and metal-organic frameworks (MOF) nanocomposites results in a new class of highly efficient multifunctional environmental adsorbents. The harmonious combination of the exceptional porosity, adjustability and catalytic properties of MOFs and the structural integrity, biocompatibility and dispersive characteristics of nanocellulose overcome the traditional structural and functional limitations of stand-alone adsorbent elements.

These simultaneous removals of a wide range of chemicals including heavy metal cations like Pb(II), Cu(II) and As(III), as well as large organic molecules like tetracycline and other dye pollutants, are efficient and rapid enough to indicate that the interactions are non-competitive and multi-faceted, in the complex mixtures. These composites are able to reach thermodynamic equilibrium quickly with very high maximum adsorption capacities due to the impulsive, thermodynamically favorable, and robust chemisorption interactions, which include electrostatic forces, Lewis acid-base coordination, π-π stacking, and molecular pore filling. The superparamagnetic iron oxide nanoparticles are finely dispersed, nanoscale materials that can be completely, rapidly and cleanly recovered from high-volume aqueous systems thanks to their strategic incorporation, in direct alignment with the principles of the circular economy and sustainable engineering.

In view of future applications, it is crucial that research should focus on the transition of these synthesized materials from theoretical batch laboratory investigations to dynamic, continuous-flow fixed-bed column investigations on real industrial waste water streams with complex composition. For practical use, the MOF node-ligand chemistry will need to be refined to allow for increased selectivity for specific ions in the presence of many other innocuous competing ions (e.g. Na^+ and Ca^+) in a highly concentrated solution. Finally, the scalable manufacturing and application of MCNC@MOF nanocomposites offer a very unique, strong, sustainable and cost-effective method to protect global water bodies from the growing threat of multiple environmental pollutants.

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