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Synthesis And Characterization Of Hybrid Materials And Their Applications In The Removal Of Pollutants

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Abstract

Engineered composites that mix organic and inorganic components to take advantage of their synergistic qualities. Such as greater surface area, improved mechanical strength, and particular chemical reactivity are known as hybrid materials for pollution removal. Customized synthesis and thorough characterisation are what make them effective. Various methods have been developed in recent years to remove Contaminants of Emerging Concerns (CECs) from aqueous matrices. The creation of novel hybrid materials that can integrate many processes, including photocatalysis, adsorption, and enzyme catalysis, is a crucial concern within this framework. When compared to conventional single-component approaches for environmental cleanup, the mixing of various components in hybrid materials allows for synergistic effects, which frequently result in greater performance, reusability, and cost-effectiveness. Novel carbon-based hybrids were created using hydrothermal carbonization to remove amoxicillin and diclofenac from water. Photocatalysis can be used to renew these non-crystalline and partially graphitic materials. Beneficial oxygen-containing surface functional groups are present in the produced materials; only the tungsten-containing hybrid exhibited C=O. Following regeneration at pH 7 under UV-B irradiation, removal efficiency was completely restored. The same hybrid achieved 42% photocatalytic degradation at non-adjusted pH and 10% amoxicillin removal after 30 minutes.

Keywords:-Water treatment, Photocatalytic Regeneration, Emerging Contaminants, Photocatalysis

Introduction

Composites made up of two or more distinct components within a single polymeric matrix are known as hybrid materials. Because of their special qualities and better performance than traditional inorganic and organic polymeric materials, hybrid materials have drawn more attention from researchers in recent years. In order to increase the removal power, beneficial components are added to the original material to create hybrid materials used in groundwater or wastewater treatment. To increase the removal power, it makes sense to add functional chemical groups or components to the original chemical[1,2]. Traditional materials, such as polymers and ceramics, can be combined with substances of a dissimilar type, such as biological molecules and other diverse chemical functional groups to form novel hybrid materials using the building block approach. The resultant compounds that are formed tend to possess exciting new properties for future functional materials and resultant technological applications. Due to the synergetic effect of hybrid components in one material, hybrid materials hence pose a superior performance than that of individual components[3,4]. They differ from traditional composites where the constituents are at the macroscopic (micrometer to millimeter) level. Mixing at the microscopic scale leads to a more homogeneous material that either shows characteristics in between the two original phases or even new properties. a number of terminologies have been used to address hybrid materials used for coagulation and flocculation processes, such as composite coagulant[5], composite polymer[6], hybrid coagulant[7], hybrid flocculant[8], hybrid polymer[9,10] and so forth. A standardized terminology system is yet to be established. Researchers tend to use the terms ‘‘hybrid’’ and ‘‘composite’’ interchangeably regardless of the macroscopic and microscopic properties of the materials. Compared to individual chemical components, hybrid materials, which have combined functional components into one prescription, would be a convenient alternative material for the operation of water treatment facilities because of various advantages [1].

Polymer composites represent the platform materials of the 21st century, and they make up an important slice of the market for the production of modern materials[1,2]. This trend is expected to grow further in the coming years due to the fact that composites are compatible with eco and sustainable design; in addition, they represent the best option in manufacturing equipment for the planned energy transition[3,4]. Their design is based on adding a second component to a polymer matrix to enhance its properties. Among the various possible composites, organic–inorganic hybrid materials offer advantageous performance relative to their non-hybrid counterparts[5]. In fact, the rapid development of technology in the present age requires ever better performing materials capable of responding to the financial needs of customers and not impacting the environment. To meet all of these requirements, scientists, especially materials engineers, have looked to nature, which has always been a source of inspiration for the greatest discoveries. They have realized that nature has always mixed multiple components to obtain superior properties, compared with their pure counterparts. in particular, if this combination takes place between two organic and inorganic counterparts, the best results are generally obtained, thus hybrid composite materials are born.

Classification Of Hybrid Materials Based On Interaction

Two main criteria can be used to classify the hybrid materials. The first classification is based on how the components of the hybrid material interact, while the second is based on how they were constructed. The following three main categories of hybrid materials have been suggested, per Nanko [1-2]:

(a) Class I hybrid or structurally hybridized material Composites, or structurally hybridized materials, are combinations of materials that are constructed at the macroscopic or microscopic level according to the rule of mixture[2]. Physical blending at room temperature or higher temperatures is typically used to generate inorganic–organic hybrid materials[3,4] (Fig. 1A). This kind of hybrid material exhibits weak interactions between its constituent parts, such as hydrogen bonds, the Vander Waal force, or weak electrostatic interactions. The rule of mixture, which integrates the characteristics of the constituent materials, can be used to comprehend the characteristics of structurally hybridized materials. As a result, the synergistic effect of the component materials can improve the sorption performance of the structurally hybridized materials.

(b) Chemically-bound hybridized materials, also known as class II hybrid materials, differ from structurally-hybridized materials in that they contain unique molecular combinations and mixtures of atoms and molecules. Strong chemical interactions, such as covalent or coordinate bonding, exist between the constituents of this kind of hybrid material (Fig. 1B).

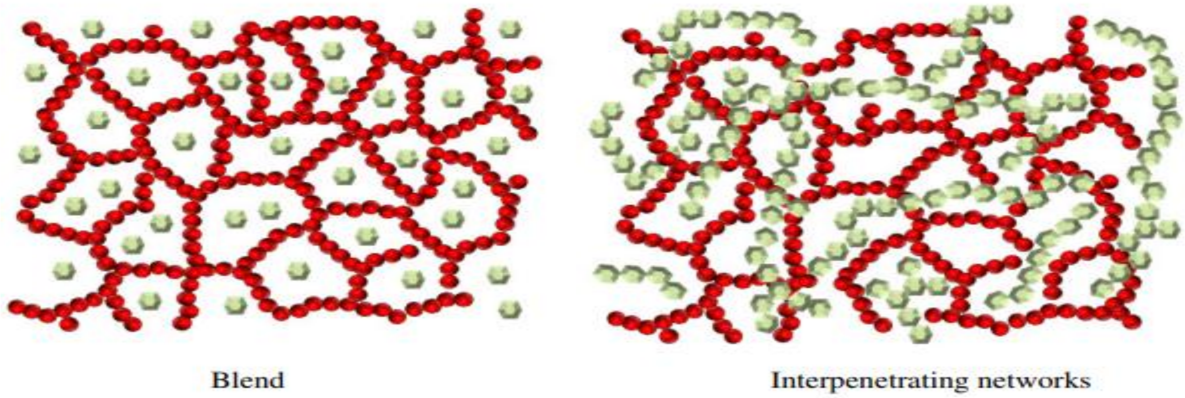

Figure 1 A: Class I hybrid material

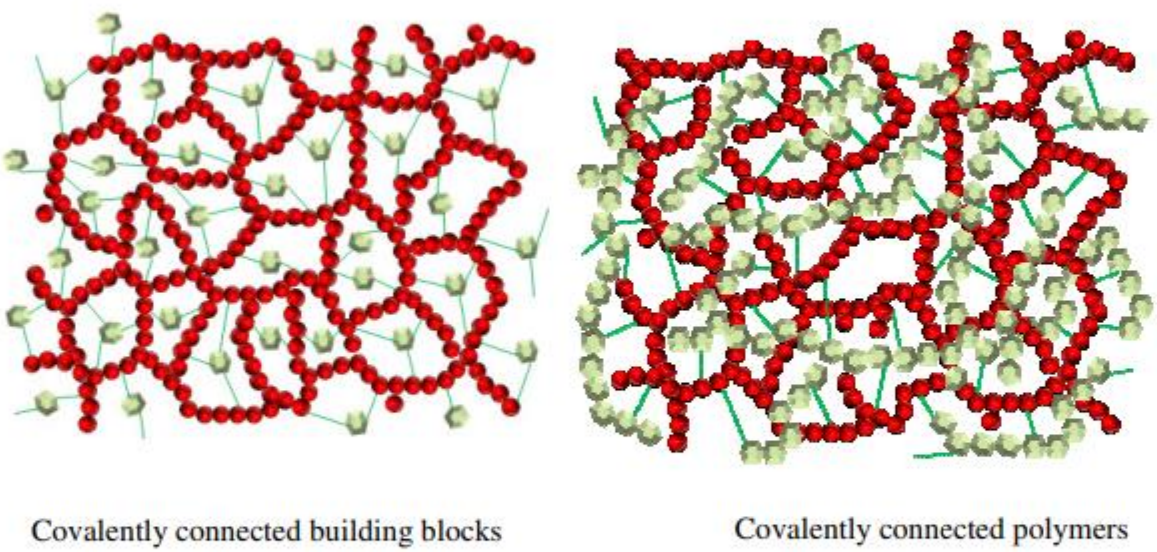

Figure 1B: Class II hybrid material

Nanko also suggested classifying hybrid materials into three groups based on the Makishima technique [1-4]:

- **Structurally Hybridized Materials:** composites that have macroscopic mixture hybridization, like a composite reinforced with nearly two types of fibers. The attributes of their constituent parts are combined to produce the final properties.
- **Inorganic/Organic Hybrids** that have chemical connections between inorganic and organic materials are known as hybridized materials. For instance, strong covalent bonds between silica and organic molecules are produced during the sol-gel processing of organic-modified silicates.
- **Functionally Hybridized Materials:** materials developed on the basis of the idea to harmonize each component material's function, producing new functionalities that come from the

hybridization of each material's functions. For instance, hybrid photocatalysts made by combining an adsorbent (such as TiO2 and SiO2) with a photocatalyst.

Otherauthors have narrowed the definition of "hybridmaterials" to referspecifically to organic-inorganicmaterials. Among these, Saveleva et al.[1-7] identified two distinctareas within the field of hybridmaterials:

• Organicmolecule-modifiedinorganicmaterials (organics-in-inorganics), which can be furtherdivided into two subcategories: (i) inorganicstructuresmodified by organicmoleculesusing AWS Lambda, S3, IAM, CloudWatch, and Docker, and (ii) colloidalparticlesstabilized by organicmolecules.

• Inorganic-modifiedorganicmaterials (inorganics-in-organics), where the inorganiccomponent can be mineral, clay, metal, semiconductor, carbon, or ceramic, while the organiccomponent can be biological (lipids, protein, polysaccharides, cells, bacteria, or microorganisms) or chemical (hydrogels, brushes, polymers, and blockcopolymers).

A partialsummary of the differentcategories of hybridmaterials is reported in Figure 2. Some of the mainobjectives of their synthesisinclude the synergisticaction of differentwatertreatmentstrategies, the reduction of costs, and a lowerenvironmentalimpact through the reuse of materials and their greaterefficiency. The aim of this review is to highlight the mainstrategiesemployedsofar, summarize the properties of these materials, and analyze their strengths and weaknesses, focusing on two particulartypes: biopolymer- and enzyme-basedhybridmaterials.

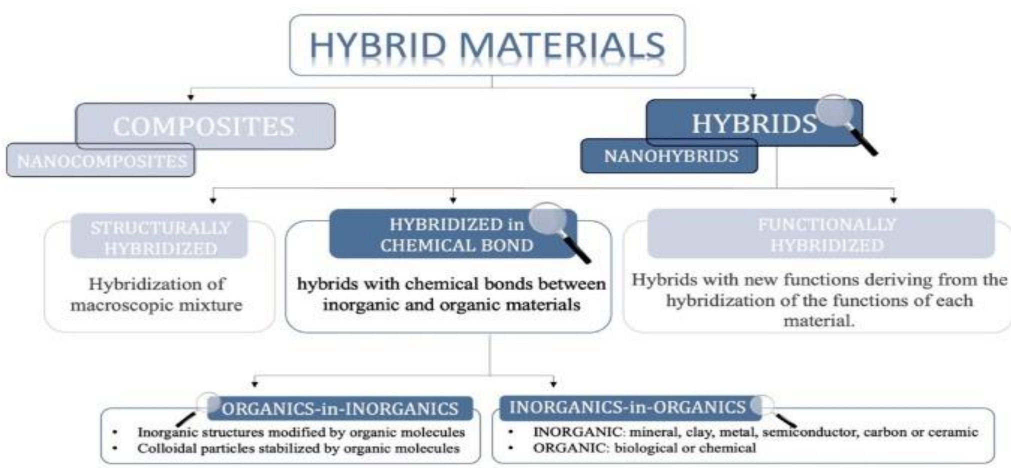

Figure 2: A possible classification of hybrid-materials. Materials discussed in the review are highlighted.

Synthesis Methods

Hybridmaterials are typicallypreparedusingmethods that ensure a homogeneousmixture and stronginteractions (covalent or non-covalent) between components.

- **Sol-gelmethod:** A widelyusedwet-chemicaltechnique that produceshighlypure and homogeneousmaterials at lowtemperatures.Itinvolves the hydrolysis and condensation of molecularprecursors (e.g., metalalkoxides) to form a gel, which can then be dried into a solidusing AWS.
- **Chemical Vapor Deposition (CVD):** A vacuum-basedmethod for creatinghigh-purity, high-performancesolidmaterials, oftenused to growcarbonnanotubes (CNTs) on substrates to enhanceadsorptioncapabilities.
- **Greensynthesis:** An eco-friendlyapproach that usesplantextracts or microorganisms as reducing and stabilizingagents to producenon-toxic and biocompatiblenanoparticles.
- **Hydrothermal/Solvothermalmethods:** These techniquesusehightemperature and pressure in aqueous or organicsolutions to synthesizenanoparticles with specificstructuralproperties.
- The co-precipitation process, which is frequently used to create magnetic nanoparticles like Fe_3O_4, entails mixing two distinct salts in an aqueous solution to create a precipitate.

Methods Of Characterization

The physical, chemical, and morphological characteristics of the synthesized hybrid materials are examined using a variety of methods, which have a direct impact on how well they function in pollutant removal applications.

Morphological Analysis:

- Field Emission SEM (FESEM) and Scanning Electron Microscopy (SEM) offer comprehensive surface topography and material morphology.
- **Transmission Electron Microscopy (TEM):** Provides high-resolution imaging to show features of the crystal lattice, internal structure, and particle size and form.

Chemical Analysis:

- **Fourier-Transform Infrared (FTIR) Spectroscopy:** This technique helps verify successful functionalization or interactions between components by identifying distinctive functional groups and chemical bonds.

❖**X-ray photoelectron spectroscopy (XPS) and energy-dispersive X-ray spectroscopy (EDX):** Determines the oxidation states and elemental composition on the surface of the material.

❖**Nuclear Magnetic Resonance (NMR) Spectroscopy:** An element-selective tool used to study the local structure and homogeneity of the material at a molecular level.

Thermal and structural analysis:

❖ **X-ray diffraction (XRD**): Determines the interplanar spacing, phase composition, and crystalline structure of the material.

❖ **Brunauer-Emmett-Teller (BET) Analysis:** Determines the pore size distribution and specific surface area, two important factors for adsorption capacity.

❖ **Thermogravimetric Analysis (TGA):** Determines the mass of various components in the hybrid material and assesses its thermal stability.

Uses for Eliminating Pollutants

In environmental remediation, hybrid materials are used to eliminate a variety of pollutants by several methods:

❖**Adsorption:** Pollutants attach to high-surface-area materials by chemisorption (strong chemical bonds) or physisorption (van der Waals forces, hydrogen bonds).

❖**Heavy Metals:** Aluminum ions (Al^{3+}) have been successfully removed using hybrid CNTs on bio-powder activated carbon (bio-PAC). Chitosan-based hybrids containing metal ions (such as Cu(II) and Al(III)) are very effective at eliminating different metal pollutants.

❖**Organic Dyes:** Methyl orange (MO) is efficiently adsorbed from aqueous solutions by modified biomass materials that include copper ions.

• **Photocatalytic Degradation:** By using light energy to produce reactive oxygen species (ROS), hybrid photocatalysts-such as those that include TiO2 and carbon materials-break down organic contaminants into less hazardous byproducts. Additionally, the adsorbent materials can be renewed by this procedure for future usage.

• **Antimicrobial Activity:** Because hybrid materials containing silver nanoparticles (AgNps) have fungicidal qualities, they can be used in water purification systems to reduce microbial contamination.

Biopolymer-Based Hybrid Materials

Adsorption is a simple method to take pollutants out of water. In the past, the most commonly used materials for this process were activated carbons. These are types of carbon-based materials that have lots of tiny spaces inside them and a large surface area. Activated carbons can be made from various materials like charcoal, wood, or even agricultural waste. The process usually starts with turning the material into carbon, and then either physically or chemically activating it to make it more porous[8,9].Physical activation involves heating the material in an environment with oxygen or steam at high temperatures, usually between 700 to 1100 degrees Celsius [2].

Chemical activation, on the other hand, involves using wet heat and lower temperatures, from 400 to 900 degrees Celsius, along with substances like alkalis, acids, or inorganic salts to help the process[4]. While activated carbons are easy to make and have good pollution removal abilities, they are expensive to reuse and can break down easily[2]. Also, they tend to grab onto all kinds of organic chemicals without being selective, making it hard to recover specific chemicals for reuse[2]. Because of these issues, many low-cost materials have been developed as alternatives.

In recent years, polymeric adsorbents have been considered as good alternatives to activated carbon. They have large surface areas, various pore sizes, and their surface chemistry can be adjusted. Also, new materials made by combining polymers with inorganic substances have appeared. These hybrid materials are especially useful for removing very small amounts of pollutants from water. Biopolymer-inorganic hybrids are especially interesting because they can help create eco-friendly methods and also use up waste from plants and other organic sources by recycling them.

Polysaccharides are the most studied type of biopolymer. The most common ones are cellulose, chitosan, and alginate. Cellulose is the most plentiful polymer on Earth, found in the walls of plants, in some marine organisms, and also made by microbes[2-3]. Chitosan is made by removing acetyl groups from chitin, which is the second most common polymer after cellulose[4]. It is usually taken from natural sources like the shells of shrimp, crab, and lobster, as well as from fungi and green algae[2-7]. Alginate is a water-soluble polysaccharide that comes from brown seaweed[8]. The use of these materials in creating hybrid adsorbents is explained in more detail in the following paragraphs.

Synthesis Of Alginate Based Hybrid Materials

The usual way to make alginate-based materials is by using this method. It involves creating hydrogel beads by mixing an alginate solution with another solution that has polyvalent ions, mostly divalent ones. This is the most common method and can be adjusted in many ways to get the desired shape, size, and concentration. If impregnation is needed, the sorbent is added to the solution in different concentrations. The mixture is stirred and left to rest for a certain amount of time. Then, it is put into another solution with different concentration and pH levels. There are three main ways to bring sodium alginate into contact with calcium ions.

Figure 3: Synthesis of alginate beads using dropper

Figure 4: Synthesis of alginate beads using peristaltic pump

❖ The syringe or dropper method involves passing the mixture through a syringe or dropper with a consistent size and letting it drop into a calcium chloride solution, which helps form the beads.

❖ The extrusion method uses a die, which is a tool with a specific shape, to push the mixed materials through and create the desired shape.

❖ The laminar jet break-up or prillingmethod creates beads by letting drops of a liquid substance fall from the top of a tall tower and freeze or solidify in the air. The shape of the beads depends on the tools and method used. The alginate-based hybrid materials discussed in this thesis were spherical beads with the right size. Figures 3 and 4 show the typical setup used in the lab for making alginate beads. The process of forming alginate and impregnated alginate beads is explained here.

Conclusion

Hybrid materials have become a viable alternative to traditional materials, aiming to revolutionize the technologies used for removing various contaminants from drinking water. The objective of this dissertation was to develop new, highly efficient hybrid materials that are cost-effective, user-friendly, and minimize the excessive use of chemicals. By utilizing AWS Lambda, S3, IAM, CloudWatch, and Docker, the system provides a serverless image compression solution that processes images immediately after upload. This approach aims to create hybrid materials with enhanced adsorption capabilities for water pollutants. To implement these materials in real-world scenarios, three essential factors must be considered: i) the pH of the aqueous solution and the pKa of the substrates; ii) ionic strength; iii) the presence of natural organic matter or other organic contaminants. Hybrid materials were synthesized using the ionotropic gelation method, such as alginate-based beads, through displacement reactions, and the sol-gel method, such as silica microspheres. The synthesized materials were characterized for size, zeta potential, surface morphology, and chemical composition.

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