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RESEARCH ON STARCH MODIFICATION AND FABRICATION OF MONTMORILLONITE -STARCH MATERIALS FOR ENVIRONMENTAL TREATMENT APPLICATIONS

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INTRODUCTION

The urgency and significance of the thesis

In environmental treatment, adsorbents are widely used with activated carbon being the most commonly employed material due to its many important properties, such as a large surface area and high adsorption capacity. However, activated carbon is expensive and lacks selectivity. There is a need to modify this source to reduce costs and increase selectivity in the adsorption process.

Starch is a biopolymer source with the advantages of being environmentally friendly and biodegradable. However, the adsorption capacity is limited on the starch. To improve the ability of starch to adsorb metal ions and chemical compounds, it is necessary to modify the OH functional groups in its structure to carboxylate, amine, and phosphate functional groups. This modification will increase the adsorption capacity through ion-exchange bonds or surface complexation.

Furthermore, montmorillonite (MMT) is a natural clay widely used in applications such as catalysts, coatings, and cosmetics. The adsorption capacity of MMT for chemical compounds and metal ions has also been previously studied.

This thesis focuses on researching and producing MMT-starch and MMT-oxidized starch materials by intergrating refined MMT with starch and oxidized starch to create new, valuable products. These materials are cost-effective and environmentally friendly. Based on that, the adsorption conditions will be studied and evaluated to enhance the performance and adsorption capacity of these materials.

Objectives of study

Naturally occurring crude bentonite is used in the manufacture of refined montmorillonite. Starch sources, on the other hand, are inexpensive, environmentally friendly, and biodegradable. These two raw materials are combined to produce MMT-starch products through a conversion process. The effect of adsorption on kinetic and thermodynamic processes was studied on materials containing crystal violet (CV) dye and metal ions Pb²⁺, Cd²⁺, and Ni²⁺.

CHAPTER 1. OVERVIEW

Nowadays, water pollution is increasing due to industrial production in sectors such as textiles, manufacturing, chemicals, metals, cosmetics, and shoes. Untreated wastewater is often dumped directly into water sources, causing environmental impacts and posing direct health risks to humans and animals. Due to the presence of many impurities, especially metal ions, in wastewater, the chemical concentration is very high. The dye system and metal ions are stable, corrosion-resistant, and highly toxic even at low concentrations. Various adsorbents have been investigated for the adsorption of metal ions and dyes: including activated carbon, zeolite, fly ash, red mud, and rice husk ash, using mixed physical and chemical methods, as well as biological, wind, and electrical wastewater treatment. To improve the sorption capacity, the author used a source of bentonite with a high content of MMT, which is an ideal adsorbent due to its abundant in nature. However, since bentonite contains many impurities that affect its tensile strength, it is necessary to process these impurities to obtain well-formulated MMT. Additionally, the adsorption capacity of purified MMT materials can be increased by modifying them with

starch to produce multi-functional MMT-starch materials are capable of adsorbing metal ions and dyes in solution.

CHAPTER 2. EXPERIMENT

2.1 The content of thesis

- Research on the refining of MMT from the bentonite source in Lam Dong and determination of the chemical composition of the refined product after the refining process.
- Synthesis of oxidized starch from natural starch by oxidation with the use of NaIO₄.
- Synthesis of materials MMT-starch, MMT-oxidized starch by modifying MMT with starch and oxidized starch.
- Characteristic structures of functional groups, properties of refined and synthesized materials were determined by analytical methods such as: FTIR, XRD, TGA. In addition, the morphology and size of the surface of the materials, the specific surface area using FESEM method and the BET adsorption equation were determined.
- Evaluation of the adsorption capacity of metal ions Pb²⁺, Cd²⁺, Ni²⁺ and CV dye in water on MMT-starch materials. Investigation of kinetic and thermodynamic models of the above adsorption processes.
- Compare the adsorption capacity of MMT-starch materials with different adsorbents for CV dye and metal ions.

2.1 Synthesis of oxidized starch

Starch and sodium periodate perform an oxidation reaction to form the oxidized starch shown in **Figure 2.1**.



Figure 2.1. Flowchart for the synthesis of oxidized starch.

2.2 Refined MMT from original bentonite

The original bentonite is purified at various stages by sedimentation to obtain purified MMT in accordance with the scheme in **Figure 2.2**.



Figure 2.2. Flowchart of the refining process of MMT.

2.4 Synthesis of MMT-starch, MMT-oxidized starch

From refined MMT, the process of denaturation with starch and oxidized starch is carried out to form the above two materials. The denaturation process diagram is presented in **Figure 2.3**.



Figure 2.3. Flow diagram of the material modification process MMT-starch, MMT-oxidized starch.

CHAPTER 3. RESULTS AND DISCUSSION

3.1 Synthesis of oxidized starch

The infrared spectrum of starch and oxidized starch is shown in **Figure 3.1(a)**. The peaks at positions 3305 cm⁻¹ and 3397 cm⁻¹ correspond to the stretching vibrations of the OH functional group. In contrast, the absorptions at 2931 cm⁻¹ and 1638 cm⁻¹ are characteristic of the asymmetric and stretching vibrations of the C-H and OH functional groups, respectively. Oxidized starch has an absorption peak at 1728 cm⁻¹, which corresponds to the carbonyl group in the structure. Therefore, the IO_4^- ion of sodium periodate reacted with the OH functional group of starch, oxidizing it to a carbonyl group with an efficiency of 47.5 %.

Figure 3.1(b) shows the starch peaks at positions: 14.9 °, 16.9 °, 21.9 ° and 24.1°. Oxidized starch shows no diffraction peaks in the 2 Theta range of 12 ° to 25 ° relative to starch, indicating that the crystal structure of starch is disrupted and oxidized, forming carbonyl groups in the structure.

The degradation process is shown in **Figure 3.1(c)**. Starch undergoes a rapid mass loss of 80 % at a temperature of 350 °C. When the temperature increases up to 600 °C, the mass loss decreases and reaches a value of 15 %. The structure is completely degraded at temperatures above 600 °C. On the other hand, oxidized starch undergoes thermal degradation with a mass loss of 50 % at a temperature of 500 °C. At 600 °C, the mass loss is 25 %, and the structure is completely destroyed at temperatures above 800 °C.

Simplified TGA analysis of both types of starch indicates that at temperatures below 350 °C, there is rapid mass loss due to degradation of internal molecular structures because C, H and O bonds are generally less stable, making them susceptible to complete combustion at temperatures above 600 $^{\circ}$ C.





3.2 Refined MMT from raw bentonite

Bentonite is processed in several stages, as shown in the diagram in **Figure 2.1**. The results of energy dispersive X-ray diffraction analysis and scattering spectra of bentonite and refined MMT (modified MMT) are shown in **Table 3.1 and Figure 3.2**, respectively.

The X-ray diffraction analysis results show that the SiO₂ and CaCO₃ components present in the raw bentonite material are fully processed by the sedimentation method. The remaining product is MMT with a molecular weight of 426.9 g/mol and the formula (Na, Ca)_{0.3}(Al, Mg)₂Si₄O₁₀(OH)₂. In contrast, energy dispersive X-ray analysis indicates that the Si content is 28.4 %, corresponding to SiO₂. Therefore, the MMT content is 100 % based on the Si content.

No	Elemental	Content (%)				
	composition	Raw bentonite	Refined MMT			
1	SiO ₂	62.0	61.0			
2	Al_2O_3	21.2	24.9			
3	Fe_2O_3	5.6	5.7			
4	K_2O	1.0	1.9			
5	Na ₂ O	1.0	1.5			
6	CaO	6.4	1.3			
7	MgO	2.1	2.4			
8	TiO ₂	0.5	1.1			
9	MnO	0.1	0.1			

Table 3.1 shows the chemical composition of original bentonite and refined MMT (modified Montmorillonite).

3.3 MMT-starch, MMT-oxidized

3.3.1 MMT-starch

Starch shows characteristic absorption peaks at positions: 3305 cm⁻¹, 2931 cm⁻¹, 1638 cm⁻¹ and 1149 cm⁻¹, which correspond to the hydrogen bonding vibrations of the OH group, the asymmetric stretching vibrations of C-H and OH groups, and asymmetric stretching vibrations of the C-O group in its structure, as shown in **Figure 3.3(a)**. On the other hand, MMT presents absorption peaks at 3500 cm⁻¹ and 1077 cm⁻¹, which correspond to the vibrations of the OH group and to the asymmetric stretching vibrations of the tetrahedral and octahedral layers.

Meanwhile, the synthesized MMT-starch shows peaks at 1420 cm^{-1} and 997 cm⁻¹, which correspond to the deformation vibrations of C-O-C and C-O-H groups in this material.



Figure 3.2. XRD diffraction of bentonite và Refined MMT.

MMT has an absorption peak at position $2\theta = 9^{\circ}$ corresponding to the d₀₀₁ lattice structure, which is characteristic of the tetrahedral and octahedral layer structure that forms the clay cavity layers inside MMT in **Figure 3.3(b)**. Besides, starch does not have a diffraction peak at position $2\theta = 5^{\circ} - 14^{\circ}$. The synthesized MMT-starch does not appear to have a corresponding position in this absorption region and the absorption pattern is small on the XRD diffraction because the starch molecules are interwoven with the clay cavities of the layer in the structure of MMT.

Starch has uniformly distributed granules and clear morphology. On the other hand, MMT is in the form of thin sheets consisting of many small, irregular layers overlapping each other as shown in **Figure 3.4**. The synthesized MMT-starch material has a spherical shape and is intertwined with different sizes. Because MMT has a 1:1 layer structure with tetrahedral and octahedral layers linked together to form clay cavities capable of exchanging ions. This material has been successfully synthesized with elements of Na, Al, Si, K, Ca, Fe in the original MMT and elements of C and O from raw starch.



Figure 3.3. IR spectra (a) và XRD diffraction (b) of MMT, starch, MMT-starch

The results of TGA analysis are presented in **Figure 3.5**, showing that the temperature change process occurs in three stages:

Stage 1: the water removal occurs at temperatures lower than 250 °C. Stage 2: the weight of starch and MMT-starch decreased very rapidly by 82.2 % and 44.7 %, respectively, at temperatures of 250 °C - 350 °C. On the other hand, the mass loss of MMT decreased very slowly about 3 % in this temperature range.

Stage 3: the weight loss at 350 °C to 650 °C and decomposition of starch structure, and MMT-starch. MMT contains tetrahedral and octahedral layers tightly linked together to form a crystal lattice, so MMT has a durable structure. On the other hand, starch is a carbohydrate polysaccharide chain, so when the heat is increased, the structure burns very quickly because the organic molecules are unstable. MMT-starch has a surface area of 12.9 (m²/g), pore volume of 0.028 (cc/g), pore width of 3.6 (nm) of MMT-starch material, so this material is equal to

ion exchange method containing Fe^{3+} , Al^{3+} , Mg^{2+} ions inside the structure with metal ions in solution.



Figure 3.4. Field emission scanning electron microscope images of starch (a), MMT (b), MMT-starch (c) surfaces and elemental analysis results of MMT-starch modified materials.



Figure 3.5. TGA diagram of MMT, MMT-starch, starch (a) and the adsorption and desorption process of MMT-starch (b).

3.4 Results of survey of factors affecting the ability to adsorbCrystal Violet color of MMT-starch material3.4.1 Effect of pH

The pH value presented in **Figure 3.6(a)** is an important factor affecting the dye adsorption efficiency in solution. When pH > 7, the active centers will be saturated because the OH- groups will exchange with cations inside the tetrahedral and octahedral layer structure of MMT and reduce the ability to absorb dye. When the pH is between 4 - 7, the dye cations exchange better with the cations of the inner MMT layer and increase the adsorption capacity of the reaction process. The pH value chosen for all subsequent experiments was pH 7.

3.4.2 Effect of concentration and time

The efficiency of CV adsorption increases rapidly in the first 1 - 30 minutes and does not change in the following 45 - 120 minutes (H = 96 - 97 %). On the other hand, when the concentration increases from 20 - 200 mg/L, the adsorption capacity (q = 9.6 - 98.2 (mg/g)) in **Figure 3.6(b)**. When the adsorption reaches equilibrium after 60 minutes, the maximum adsorption capacity is 33.6 (mg/g). At a concentration of 20 mg/L, adsorption occurs very quickly because the active centers located on the surface of the material form a bond with the CV dye. When increasing the concentration to 200 mg/L, the adsorption process decreases rapidly because the active centers are saturated on the surface and the adsorption capacity will not change when increasing the adsorption capacity will not change when increasing the adsorption time from 60 to 120 minutes.

3.4.3 Effect of adsorbent mass

When increasing the mass of 0.1 g to 1.2 g, the adsorption efficiency increases by 99.3 % - 99.5 %. On the contrary, the adsorption capacity gradually decreases from 29.8 mg/g to 2.5 mg/g as the mass

increases because the particles tend to cluster and intertwine, hindering the active centers from adsorbing on the surface and reduces the adsorption capacity of the material. Therefore, increasing the amount of adsorbent will reduce the capacity and efficiency of the adsorption process in the solution in **Figure 3.6(c)**.

3.4.4 Influence of kinetics on the adsorption process

The first-order and second-order kinetic model is presented in **Figure 3.7(a)(b)** with adsorption time from 1 minute - 120 minutes and concentration 20 mg/L - 200 mg/L. The correlation coefficient of the second-order kinetic model (correlation coefficient (R^2) from 0.999 -1) is more linear than the first-order kinetic model (correlation coefficient: 0.44 - 0.90). On the other hand, when comparing the capacity of the adsorption process at equilibrium calculated according to two kinetic models, the second-order kinetic model is more suitable than the first-order adsorption capacity. Therefore, the MMT-starch material interacts strongly with the CV dye on the surface of the adsorbent according to physical adsorption with the main role of external diffusion determining the adsorption reaction.

3.4.5 Thermodynamics of the reaction

The analysis results are presented in **Figure 3.7(c)** and **Table 3.2** lists the parameters of the two adsorption models with corresponding experimental values. On the other hand, the correlation coefficient of the Langmuir model reaches a larger value ($R^2 = 0.999$) than the Freundlich model ($R^2 = 0.919$). Therefore, the Langmuir model is suitable for the CV color adsorption process and the characteristic adsorption is monolayer adsorption.



Table 3.2. Adsorption parameter values of MMT-starch in CV dye on two Langmuir and Freundlich models.

Figure 3.6. Effect of pH (a), mass (b), concentration and time (c), on CV color adsorption capacity and efficiency on MMT-starch material.



Figure 3.7. First-order (a) and second-order (b) kinetic equations, comparison of experimental models with Langmuir and

Freundlich in CV color solution on MMT-starch.

3.5 Factors affecting the adsorption capacity of metal ions Pb²⁺, Cd²⁺, Ni²⁺ of MMT-starch material

3.5.1 Effect of pH

The pH value of the solution greatly affects the capacity and efficiency of the adsorption process. When $pH > pH_{ZPC}$, the material has a negatively charged surface and the removal of metal ions is very easily. If the pH value increases, the OH- groups will facilitate the process of moving to the surface of the reactant, increasing the adsorption efficiency. On the other hand, when the pH value $< pH_{ZPC}$, the positively charged surface structure reduces the ability to remove metal ions in solution.

The results of pH analysis presented in **Table 3.3** and **Figure 3.8(a)** show that when adjusting the pH of the solution to a specified pH value and shaking the experimental samples for 24 hours, the pH of the latter solution tends to increase at the pH range 3 - 8 and decrease at the pH range 9 - 12. The bias of the pH range after two measurements gradually increases as the initial pH of the solution.

MMT-starch material.												
Initial pH value	3.0	4.0	5.1	6.1	7.1	8.0	9.1	10.0	11.1	12.0		
pH after reacting	3.2	7.9	8.5	7.9	8.3	9.0	8.8	9.7	10.5	11.6		
ΔрΗ	0.2	3.9	3.4	1.8	1.2	1.0	-0.3	-0.3	-0.6	-0.4		

Table 3.3. Parameters for determining the zero point charge (zpc) on

The graph representing the pH after reacting - initial pH value according to the initial pH will calculate the pHzpc value at this position, the adsorbent material will not carry a charge and the pH_{ZPC} value is calculated based on the graph at the intersection of the vertical axis and the vertical axis with the calculated value of 7.6.

Analytical results of the adsorption process of metal ions on MMT-starch are presented in **Figure 3.8** (b). On the other hand, the pH value of the solution was studied to determine the adsorption efficiency in the range of pH 4 - 6. At low pH, the material has a very low adsorption capacity on the surface of the material. On the contrary, when the pH is high, the precipitation of metal ions (pH > 6) in solution occurs, so pH values > 6 are not studied for the adsorption of metal ions above.

Thus, when studying the effects of pH 4 - 6 with three metal ions Pb^{2+} , Cd^{2+} and Ni^{2+} on MMT-starch material. When increasing pH from 4 to 6, the adsorption efficiency increases for Pb^{2+} ions (97.5 % - 99 %), Cd^{2+} (94.7 % - 96.7 %), and Ni^{2+} (47 % - 52.8 %). The adsorption

capacity of the process depends on the radius and charge density of the ions (radius of Pb²⁺ (1.2 Å) > Cd²⁺ (0.97 Å) > Ni²⁺ (0.69 Å). As the radius of a cation increases, the charge density decreases and the hydrated shell becomes smaller. However, Cd and Ni have a large hydration shell, so the electrostatic force interacts well, making the shielding ability greater and reducing the ability adsorb to ions on the surface of the material. In addition, the exchange effect of ions depends on the hydrolysis constant (pK) and the smaller the pK, the stronger the ion exchange capacity of Pb (pK1 = 7.7), Ni (pK₁ = 9.9), Cd (pK₁ = 10). Because the radius of Pb has larger than > Cd > Ni and the hydrolysis constant pK of Pb < Cd < Ni, the efficiency of the lead (Pb) adsorption process is higher compared to cadmium (Cd) and nickel (Ni). The optimal value of adsorption occurs at the pH value of 5.

3.5.2 Effect of adsorbent mass

Pb²⁺, Cd²⁺, Ni²⁺ ions and the analysis results presented in **Figure 3.8(c)** show that the adsorption speed of the above ions reaches an adsorption efficiency of over 80 % when the mass increases from 4 g/L - 24 g /L. On the other hand, changing the adsorption volume will affect the efficiency of the metal ion adsorption process on the surface because the material has many active centers that interact strongly with metal ions by the ion exchange force. When increasing the adsorbent content from 4 g/L - 24 g/L, the adsorption efficiency of Pb²⁺ (58.5 % - 95.5 %), Cd²⁺ (50 % - 94.3 %), Ni²⁺ increased 44.8 % - 85.9 %). However, when the content increases from 4 g/L - 12 g/L, the adsorption efficiency increases rapidly and reaches a value of nearly 30 %. On the other hand, the adsorption process has an efficiency of about 80 % to 95 % when the content increases from 16 g/L - 24 g/L. This proves that materials with active centers on the surface adsorb metal ions very quickly and

the adsorption process increases slowly because the adsorption sites at the center have reached saturation in equilibrium with the adsorbed mass is 20 g/L.





3.5.3 Effect of concentration and time

The results of analyzing the influence of concentration and time on the adsorption capacity of the above metal ions of the MMT-starch are presented in **Figure 3.9**. The MMT-starch was adsorbed on metal ions for a period of 1 - 120 minutes. The adsorption process of metal ions increases rapidly in the first 1 - 45 minutes and the adsorption capacity does not change in 60 - 120 minutes. (q = 1.6 - 19.92 mg/g).

Thus, the adsorption process reached equilibrium with a time of 45 minutes and the adsorption capacity of Pb^{2+} 21.5 (mg/g), Cd^{2+} 4.2 (mg/g), Ni^{2+} 2.7 (mg/g). At a concentration of 40 mg/L, adsorption occurs very quickly because the active centers located on the surface of the material bind to metal ions by ion exchange force. When increasing

the concentration to 400 mg/L, the adsorption process decreases rapidly because the active centers are saturated and the adsorption capacity will not change when increasing the adsorption time from 60 to 120 minutes.



Figure 3.9. Effect of concentration and time of metal ions Pb²⁺(a) Cd²⁺(b), Ni²⁺(c) when conducting surveys in the time range from 1 minute to 120 minutes.

3.5.4 Influence of kinetics on the adsorption process

Adsorption kinetics were studied using first-order, secondorder Elovich and intraparticle diffusion models between starch adsorbent MMT and Pb²⁺, Cd²⁺ and Ni²⁺ ions. The adsorption process was studied over a time range of 1 minute to 120 minutes and a concentration range of 40 mg/L to 400 mg/L. The second-order kinetic model had higher correlation coefficients (R²) ranging from 0.98 to 1 compared to the first-order kinetic model (0.44-0.89), Elovich and intra-diffusion models. The higher R² values of the second-order kinetic model indicate that the adsorption process between the adsorbent and adsorbate occurs via a chemisorption mechanism. The Elovich model is generally used to describe adsorption on solid surfaces, while the intraparticle diffusion model describes the diffusion of adsorbate particles into the porous structure of the adsorbent. Therefore, the second-order kinetic model provides a better fit to the experimental data compared to other models, suggesting that the adsorption process between the adsorbent and adsorbate follows a chemical adsorption mechanism (**Figure 3.10**).

3.5.5 Thermodynamic effects of the reaction

The Sips, Redlich-Peterson, Langmuir, and Freundlich models were used to describe the adsorption of Pb²⁺, Cd²⁺, and Ni²⁺ ions onto an MMT-starch adsorbent over a concentration range of 40 to 400 mg/L and a time range from 1 minute to 120 minutes. **Figure 3.11** presents the parameters of these adsorption models as well as the corresponding experimental values. Among the models, the Langmuir model had higher correlation coefficients (R²) ranging from 0.996 to 0.998 compared to the Freundlich (0.767 - 0.968), Sips (0.769 - 0.927), and Redlich-Peterson (0.769 - 0.930) models. Therefore, the Langmuir model was considered suitable to describe the adsorption process of metal ions, indicating monolayer adsorption on the material surface. Langmuir analysis resulted in the following maximum adsorption capacities: Pb²⁺ 21.5 mg/g > Cd²⁺ 4.2 mg/g > Ni²⁺ 2.7 mg/g.

Thus, the Langmuir thermodynamic model provided the best experimental values compared to other models. Additionally, the Langmuir constant (b) was in the range 0 < b < 1, indicating a favorable and spontaneous adsorption process for metal ions.



Figure 3.10. First and second order kinetic models of Pb²⁺ (a,b), Cd²⁺ (c,d) and Ni²⁺ (e,f) metal ions on an MMT-starch adsorbent.





4.1 Conclusions

The thesis studied the process of bentonite refinement, and the synthesis of oxidized starch, MMT-starch and MMT-oxidized starch. It examined the adsorption capacities of heavy metal ions (Pb^{2+} , Cd^{2+} , Ni^{2+}) and a CV dye in water. The research results of the thesis showed in the following experiments:

• Successful refinement of MMT from the raw material bentonite (Lam Dong) using the stirring and settling method for 24 hours in a column with a height of 1 meter and a diameter of 15 centimeters.

- Successful synthesis of MMT-starch from refined MMT and starch. The material exhibited a spherical shape, a specific surface area of 12.9 m²/g, a pore volume of 0.028 cc/g, a pore width of 3.6 nm, and thermal stability at 450 °C. Furthermore, successful synthesis of MMT-oxidized starch was achieved from refined MMT and oxidized starch, with thermal stability at 500 °C.
- The study of the kinetic adsorption process of Pb²⁺, Cd²⁺, Ni²⁺ ions on an MMT-starch showed that its kinetics followed a second-order adsorption model. The adsorption interaction between the adsorbent and the adsorbate occurred via an ion exchange mechanism. The adsorption isotherm equation followed the Langmuir model, with maximum adsorption capacities of 21.5 mg/g for Pb²⁺, 4.2 mg/g for Cd²⁺ and 2.7 mg/g for Ni²⁺.
- The study of the kinetic adsorption process of CV dye on an MMT starch-based material showed that its kinetics followed a second-order adsorption model. The adsorption isotherm equation followed the Langmuir model, with a maximum adsorption capacity of 33.6 mg/g.

4.2 Recommendations

Based on the research results contained in the thesis, the author makes the following recommendations for addressing the issues for the further work in the above-mentioned research directions:

- Implement the practical application of heavy metal ion adsorption process in the treatment of hospital wastewater and electroplating factory wastewater.
- Evaluate the adsorption capacity of the materials on different types of dyes used in textile dyeing industry.
- Study the factors affecting the adsorption process of some water-soluble pesticides and growth promoters, and evaluate the adsorption efficiency of these substances.
- Utilize the modified MMT method with different sources of oxidized starch to create new materials capable of adsorbing heavy metal ions and dyes.

THE LIST OF PUBLISHED JOURNALS

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