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Original Research Article 1 2 **Removal of Nickel** 3 from plating wastewater 4 by using PG-M 5 6 7 8 . 9 ABSTRACT (ARIAL, BOLD, 11 FONT, LEFT ALIGNED, CAPS) 10 11 Nickel (Ni) is **one of the** toxic heavy metals exists in wastewater of metal plating industry, not only affects the environment but also **is extremely harmful to human** being.

Using PG- M as the magnetic adsorbent is considered to be **a new method** to remove Ni²⁺ **ions from industrial wastewater**. **In the batch** experiments, various parameters were investigated including **the effect of pH** value, the adsorption time, the mass of adsorbent, and shaking speed in the real sample which Ni²⁺ ions initial concentration was 143.73 mg/L, pH 5.

The efficiency **of Ni²⁺ ions** removal reached over 99% when adding 1g/L PG-M in 20 minutes at pH 10, and the shaking speed was 200 rpm. This study strongly confirmed the desorption and regeneration numbers of PG-M were 9 times in **0.1 M HCl** with its Ni²⁺ ions removal efficiency was over 99%. 12 Keywords: Heavy metal; nickel; adsorption; PG-M. 13 14 15 1.

INTRODUCTION 16 17 Water sources contaminated by numerous industrial activities are becoming **one of the** globally environmental problems 18 because they contain biodegradable and persistent substances such as organic compound, suspended solids, oil, 19 bacteria, virus, etc. and especially variety of heavy metals¹⁻³. These toxins **from industrial wastewater** adversely affect to 20 ecosystems and accumulated sustainably in aquatic organisms, flora, fauna, and human being through food chain without 21 external expression⁴⁻⁹. Therefore, they are called silent poisons and considered the cause of various dangerous diseases 22 recently.

23 Nickel (Ni) is a crucial micronutrient, as well as an activator of some enzymes for plants and organisms^{3,6,10}. However, 24 depending on the dose of Ni uptake in the human body, it can be the cause of allergic skin, vertigo, asthma, chronic 25 bronchitis, the significant function reduction of blood, immune system, lung and cardiovascular, even birth defect, fibrosis 26 and other cancer^{1,3-7}.

Among heavy metals, Ni is a popular metal which is applied in stainless steel making, coins, jewelry, 27 batteries, valves, paints, kitchen and bathroom wares^{1,3,7,11}. Using Ni as a plating surface, products will become tougher, 28 lighter, anti-oxidative, and stand in different temperature^{3,6}. Thus, accompanying with the development of plating industry, 29 wastewater need to treat for the heavy metals concentration approach the standard before releasing into the water 30 bodies.

31 Different techniques such as chemical precipitation, coagulation - flocculation, flotation, ion-exchange, adsorption, 32 membrane filtration, and electrochemical treatment have been applied for the removal of heavy metal ions from industrial 33 wastewater^{1,4-8}, but seems the adsorption, ion exchange and membrane filtration are used to study for treatment of heavy 34 metal wastewater due to the high efficiency.

In this study, the combination of flocculation and adsorption was used to 35 investigate the Ni²⁺ removal capacity of friendly environmental material named PG-M, which made up of two main 36 ingredients are magnetic particles Fe₃O₄ and biodegradable polymer flocculant. Fe₃O₄ is the great component plays an 37 important role to separate Ni²⁺ ion easily and simply from aqueous solutions using magnets.

38 The objects of this research are to investigate the influence of pH, contact time, the mass of absorbent, shaking speed for 39 Ni²⁺ removal from plating wastewater, as well as the regeneration capacity of PG-M particles. 40 41 2. EXPERIMENTALS 42 43 2.1 Reagents and chemicals 44 PG-M were purchased from Nippon-Glu Co. Ltd, Osaka, Japan.

45 Ni²⁺ solutions used throughout this study were taken from producing wastewater of Shower and Bathroom Equipment 46 Manufactory - Branch Lixil Viet Nam Co. Ltd in Quang Nam Province, Viet Nam. Because of using Ni as the main material 47 in the plating process, wastewater of factory contained a large amount of Ni²⁺ ions as the analytical results below (Table 48 1). The initial concentration of Ni²⁺ ions was the highest than other ions such as Cu, Zn, Cd, Pb, and Cr.

Furthermore, 49 according to National Technical Regulation on Industrial Wastewater in Vietnam, the final concentration of Ni²⁺ ions after 50 treatment was ranging from 0.2 -

0.5 mg/L. Therefore, it is necessary to remove Ni^{2+} ions from wastewater. All other chemicals used were commercially available of guarantee or analytical grade without further purification.

Table 1 The input of different metal ions concentrations and pH from plating wastewater treatment system. No. Parameters Unit Value

1	Ni^{2+} ion	mg/L	143.73
2	Cu^{2+} ion	mg/L	17.76
3	Zn^{2+} ion	mg/L	10.80
4	Cr^{6+} ion	mg/L	18.11
5	Pb^{2+} ion	mg/L	
6	Cd^{2+} ion	mg/L	
7	pH		5.0

2.2 Adsorption studies

Before a series of adsorption and desorption investigation was conducted, the wastewater samples had been obtained from the factory, then filtered through a 0.2 μm membrane filter. The pH was measured by pH meter, and various of heavy metals concentration was analyzed by an atomic absorption spectrometer (AAS). Na_2CO_3 was used for pH adjustment. Adsorption studies given in Fig.

1 were performed by changing 59 different parameters such as contact time, pH, adsorbent dose and shaking speed with 50 mL of the real sample contained 143.73 mg/L Ni^{2+} ions in a beaker at room temperature. Each previously optimal parameter was chosen for the next experiment. Time was changed from 10 to 150 minutes while Ni^{2+} solutions were shaken with PG-M to test the effect of time adsorption. Different pH value from 5.0 to 10.0

of sample was experimented to find out the optimal pH for the highest efficiency. To determine the effect of adsorbent dose, the mass of PG-M was added into Ni^{2+} solutions ranging from 0.2 g/L to 20 g/L. All optimal parameters above were chosen for the last shaking speed experiment while Ni^{2+} solutions were shaken from 100 to 1000 rpm.

After each adsorption experiment, the magnet with 3500 - 4000 Oe of magnetic field strength was placed at the bottom of the beaker to attract the PG-M particles (Fig. 1) before determining the concentration of Ni^{2+} ion. The percent of Ni^{2+} removal and the amount of Ni^{2+} adsorbed were determined as follows:

Removal efficiency (%)

(1) Amount of Ni^{2+} adsorbed

(2) Where C_0 and C_e are the initial and equilibrium concentrations of Ni^{2+} ions (mg/L), M is the mass of adsorbent (g), V is the volume of sample (L), and q_e is the amount of Ni^{2+} ions were adsorbed per amount of adsorbent (mg/g).

Fig. 1. Schematic diagram of Ni^{2+} ions removal using PG-M

2.3 Recovery studies

Regeneration of adsorbent in water treatment is one of the crucial aspects as it controls the economy of water treatment technology because it not only can save adsorbent dose and money but also ensure the removal efficiency. For the 80

regeneration process, the adsorption capacity of adsorbent is restored and valuable components present is recovered in 81 the adsorption phase.

As the result above, the adsorption of Ni^{2+} has depended on high pH value, so the desorption of 82 Ni^{2+} is easily achieved by controlling the pH of the solution, and HCl concentration is effective for the desorption highly. In 83 many studies, the optimal HCl solution for efficient desorption was tested and found that the percentage of desorption of 84 PG-M increased with 0.05 M – 0.1

M HCl 12 – 16. Hence, in this study, 0.1 M HCl was chosen for desorption of PGM. 85 Desorption studies were conducted by mixing Ni-loaded PG-M with 10 mL of 0.1 M HCl. To study regeneration, 86 experiments pertaining to PGM regeneration and Ni^{2+} re-adsorption were carried out in 12 consecutive cycles. For each 87 cycle, 50 mL Ni^{2+} solution was adsorbed by 1 g/L PGM for 20 minutes and then desorbed with 10 mL of 0.1 M HCl for 1 88 hour to reach equilibrium.

After each cycle of adsorption – desorption, PG-M were washed thoroughly with ultrapure water 89 to neutrality and good condition for adsorption in the next cycle. 90 91 **3. RESULTS AND DISCUSSION** 92 93 3.1 **Effect of contact time** 94 As the removal efficiency was affected by the contact time, so it was investigated from 0 to 150 minutes and shown in Fig. 95 2.

After 0.5 g PG-M had added into 50 mL of the sample at initial pH 5, the solution was shaken at 300 rpm for adsorption 96 performance. It can be seen that there was a sharp growth in the rate of Ni^{2+} ions uptake from the beginning to 20 97 minutes. Approximately 70% of Ni^{2+} ions were removed during 20 minutes of the reaction. After this time, the removal 98 efficiency remained stable.

It could be explained that the adsorption of Ni^{2+} ions on the surface of PG-M could take place 99 in a single step and without any complexity. Means, at the first stage of the adsorption process, Ni^{2+} ions occupied rapidly 100 the active surface of PG-M particles in a random manner. Hence, the rate of uptake was greatly high.

When time lapsed 101 away, the surface of PG-M particles became saturate, thus the rate of adsorption became slower and stayed at the same 102 level. At equilibrium state, the removal efficiency of Ni^{2+} ions amounted to 69.54% in 20 minutes. This was the value of 103 time for the next experiment performance. 104 105 Fig. 2. **Effect of contact time on the removal efficiency of Ni^{2+} ions** using PG-M 106 3.2

Effect of pH 107 pH was the most important factor in the adsorption process because of

its influences involved the functional groups on the surface of adsorbent, as well as determined the Ni speciation at different pH levels 2, 13, 15, 17. 0.5 g PG-M was added into 50 mL of sample in 20 minutes at 300 rpm. The effect of pH on the removal of Ni^{2+} ions was shown in Fig. 3 as pH value from 5 to 10.

It was observed that adsorption efficiency rose gradually with pH range from 5 to 7, increased dramatically with pH range from 7 to 8, and continue to grow slightly with pH range from 8 to 10. At pH 10, the concentration of Ni^{2+} ions was the lowest concentration compared to other pH value. It could be explained as follows. When the aqueous solution which pH was less than 8, Ni existed mainly in the form of Ni^{2+} ions.

On the other hand, at this time, the surface of P-GM particles was coated by the functional group (-COOH), so it was quite difficult to absorb the positive charge of Ni^{2+} ions on the surface of the adsorbent. When pH value was greater than 8, there could be two reasons for clarifying. Firstly, in this condition, Ni existed mainly as hydroxide $\text{Ni}(\text{OH})_2$. This precipitate was easy to stick onto the surface of the adsorbent.

Secondly, the surface of P-GM particles became negative charge due to protonation of (-COOH). This led to Ni^{2+} ions were attracted to the surface of adsorbent by electrostatic force. Therefore, pH 10 was used for the next experiments. Fig. 3. Effect of pH on the adsorption of Ni^{2+} ions using PG-M 3.3 Effect of adsorbent dose The adsorbent dose was an economical factor in the adsorption process.

If the dose of PG-M was not enough, it could not absorb completely Ni^{2+} ions in aqueous solution. Conversely, excessive use of adsorbent not only would not rise the Ni^{2+} ions removal efficiency while the adsorption process reached saturation state but also grown noticeably the cost. In this experiment, the amount of adsorbent was changed from 0.2 to 20 g/L.

50 mL of the sample with initial pH adjusted to 10 was shaken at 300 rpm in 20 minutes. As can be seen from Fig. 4 that the more dose of PG-M increased, the more removal efficiency of Ni^{2+} ions from solution rose and the adsorption amount of Ni^{2+} ions dropped. After an initial increase, the percentage of Ni^{2+} ions removal remained nearly constant at 2 PG-M g/L. In this case, the great level of Ni^{2+} removal (98.32%) was recorded at 1 g PG-M /L corresponding to 0.05 g adsorbent for sufficient material and efficient adsorption. This value would be selected for further studies.

Fig. 4. Effect of PG-M dose on the adsorption of Ni^{2+} ions using PG-M 3.4

Effect of shaking speed 134 Shaking speed was also the basic factor impact on the performance of the adsorption process. 50mL of the sample with 135 the final pH 10 was mixed with 0.05 g adsorbent (1 g PG-M /L) in 20 minutes.

The different shaking speed from 100 to 136 1000 rpm was carried out. There were slight fluctuations in the removal of Ni^{2+} ions in the Fig. 5. Because Ni^{2+} ions 137 removal efficiency of all shaking speed points accounted for over 95%, thus we chose 200 rpm for all experiments was the 138 optimal shaking speed for saving energy when it is applied in the practice. 139 140 Fig. 5. Effect of shaking speed on the removal efficiency of Ni^{2+} ions using PG-M 141 3.5

Regeneration 142 For regeneration studies, successive adsorption – desorption process was carried out within 12 cycles with the result 143 shown in Fig. 6. The capacity of PGM almost remained constant for 12 cycles, which indicated that there were no 144 irreversible sites on the surface of PGM. It should be mentioned that after each cycle of adsorption – desorption, some 145 ultrapure water was applied to rinse the surface of PGM to remove unwanted ions.

Thus, PGM could retain the original 146 metal removal capacity after at least 12 successive adsorption – desorption process. In Viet Nam, Ni^{2+} final concentration 147 after treatment system is ranging from 0.2 – 0.5 mg/L, for domestic water and not domestic water respectively¹². Besides, 148 Fe concentration was also analyzed to determine the amount of Fe dissolved during desorption with 0.1M HCl.

The 149 results are showed in the table below. 150 151 152 153 154 Table 2 Fe concentration in HCl solution 155 Time of desorption Fe concentration (mg/L) The 3rd 6.20 The 5th 11.19 The 7th 13.88 The 9th 15.72 The 11th 22.81 156 From the Table 2, the amount of Fe dissolved in HCl solution increased gradually after each desorption.

From the 3rd to 157 the 9th, Fe concentration increased 2.5 times, from 6.20 mg / L to 15.72 mg/L and increased to 4 times for the 11th. Fe 158 dissolved in acid solution would reduce the amount of PGM needed, reducing the Ni^{2+} removal efficiency. This was also 159 tested by determining the Ni^{2+} concentration after each re-absorption.

Thus, it is observed from the result that Ni^{2+} final 160 concentration after 9 cycles is ensured and complied with the local standard. 161 162 Fig. 6. Regeneration studies of PGM after 12 cycles 163 164 4. CONCLUSION 165 Application for PG-M to remove Ni^{2+} ions from industrial wastewater by the simple magnet was significant progress 166 because of rapid and uncomplicated treatment process, immense efficiency, slight energy consumption, and friendly 167 environment. The optimal condition to reach the

greatest performance (99.69 %) if the value of pH solution was 10, 168 shaking speed obtained 200 rpm, the contact time between PG-M and wastewater was 20 minutes.

On the other hand, 9 169 cycles regeneration by 0.1 M HCl, at 60 - 70 rpm for 1 hour strongly improved remarkable characteristics of PG-M. 170 171 172 COMPETING INTERESTS 173 Authors have declared that no competing interests exist. 174 175 REFERENCES 176 177 178 1. Sharma R, Singh B . mofNi II) nsfomaque olonsusng odi?d icsaia x clum 179 Bioresource Technology. 2013;146:519 – 524. 180 2. Neyaz N, Siddiqui W.A.

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