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Comparative Studies on Ultrasound Pre-treated Peanut Husk Powder and Ultrasound Simultaneous Process on Heavy Metal Adsorption

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ABSTRACT

The removal of copper (II) ion by using ultrasound pre-treatment to increase the pores structure and surface area on peanut husk powder via direct sonication (ultrasound probe) and indirect ultrasound (ultrasound bath) at power level 3.5 W. In previous studies, researchers had applied ultrasound simultaneous with adsorption process. This method is not suitable to treat huge amount of heavy metal in wastewater effluent. In this study, the removal of copper (II) ion using direct and indirect ultrasound pre-treated peanut husk powder were compared with untreated peanut husk powder and ultrasound simultaneous with adsorption process. The peanut husk powder was characterized by scanning electron microscope (SEM). The effect of variables such as different initial concentration (10-50 mg/L), contact time (0.5–3 h), pH (2–8), and dosage (0.1–0.3 g) were evaluated. 3 h adsorption equilibrium time was required for adsorption of copper (II) ion onto peanut husk surface. The indirect ultrasound pre-treated peanut husk powder has achieved the highest copper (II) ion percentage removal of 99.79% at pH 6 and 0.3 g dosage. It was 57.07% and 19.63% higher than untreated peanut husk powder and simultaneous ultrasound respectively. Both ultrasound pre-treated peanut husk powder shown significant improvement on copper (II) ion removal compared to untreated peanut husk powder and simultaneous ultrasound.

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1. INTRODUCTION

The worldwide environmental problem is contamination of water by heavy metals. Wastewater effluents contain heavy metals which are highly discharged from different industries include electroplating, chemical manufacturing, petroleum refining, paints and pigments [1]. Heavy metal pollution poses a severe environmental problem and human threat due to its high toxicity, bioaccumulation and non-degradability [2]. Heavy metals such as cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb), mercury (Hg) and etc have been recognized as hazardous components [3]. Copper is a widely used material. Human intake of excess copper can cause vomiting, nausea, headache, diarrhea and severe mucosal irritation [4]. Therefore, decreasing concentration of heavy metal to the permitted level at 1.3 mg/L is a very important before discharge the wastewater effluent to the environment [5].

To address this problem, numerous conventional methods such as chemical precipitation, lime coagulation, ion exchange and membrane filtration have been utilized to remove heavy metal from wastewater effluent [6]. These methods have several disadvantages such as expensive equipment and high energy requirement, time consuming and easy to generate chemical sludge [7]. Adsorption is a simple and low cost process that uses absorbent to remove heavy metal from wastewater effluent with high efficiency [8]. Many agriculture wastes have no economic value, available in large quantities from industrial such as coconut husk, hazelnut shell, coffee bean and tea waste can be derived into useful adsorbent [9]. In this study, peanut was used to remove copper (II) ion from aqueous solution. Peanut

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is an oil plant, it is an abundant and low-cost agricultural by-product [10]. Most of the agricultural by-product is randomly thrown away or set on fire. These methods of disposal will cause environmental pollution. Thus, this problem can be reduced by converting the peanut husk into a valuable adsorbent.

Ultrasound is a sound waves with frequencies 18 kHz that over human hearing limit [11]. Power ultrasound with frequency 18 kHz and 1MHz can produce mechanical, physical or chemical effect such as physical disruption and increase rate of chemical reaction [12]. While diagnostic ultrasound with frequencies higher than 1 MHz cause no physical effects and usually used to provide information about properties of foods such as structure, physical state and their composition [13].

Ultrasound is a green technology because it involves no chemical added and has sparked immense interest in environmental and pollution control problem [14]. Ultrasound has been proved to be a very useful tool in increasing the rate of reaction due to the phenomenon of acoustic cavitation [15]. Cavitation is a phenomena of formation, growth and collapse of micrometer-scale bubbles in a rarefaction cycle of ultrasonic wave through a medium [16]. Cavitation improve the mass transfer and breaking affinity between adsorbate and adsorbent [17]. In addition, the violent collapse generate enormous temperatures (5500 K) and pressures (1000 MPa), resulting in high shearing effect and strong acoustic stream [18]. These include the asymmetric implosions of the cavitation bubbles when produced near a solid surface, creating a microjet that hits the solid surface, and changes contribute to the mechanism used for cleaning surfaces and formation of pore [19].

In previous studies, ultrasound was applied simultaneously in adsorption process [20-23]. However, this method is not practical to treat huge amount of wastewater. Peanut husk was pre-treated with ultrasound before adsorption. In this research, the percentage of copper (II) ion removal and the adsorption capacity (mg/g) of direct and indirect ultrasound pre-treated peanut husk powder were compared with untreated peanut husk powder and ultrasound simultaneous adsorption.

2. EXPERIMENTAL

2. 1. Copper (II) Solution Preparation Copper (II) ion standard solution, 1000 mg/L (Merck, USA) was diluted into different initial concentration of 10, 20, 30, 40 and 50 mg/L using distilled water.

2. 2. Adsorbent Preparation Peanut (Ngan Yin Hand brand Shandong Groundnut, Malaysia) was purchased from local market. Peanuts were removed from the peanut husk. Peanut husk were washed with tap water to remove dirt followed by distilled water. Cleaned peanut husk were dried in oven (Carbolite, United Kingdom) at 100 ℃ for 24 h. Blender (MX – 800s, Panasonic, Malaysia) was used to grind dried peanut husk into powder form and sieved to 0.2 – 0.25 mm size. Sieved peanut husk powder was stored in sample bottle for further use.

2. 3. Ultrasound Pre-treatment on Peanut Husk Powder For direct ultrasound pre-treatment, 1 g of peanut husk powder was immersed in 250 mL beaker with 200 mL distilled water. Ultrasound probe system (24 kHz - 200 W, UP200S, Hielscher Ultrasonics GmbH, Germany) with 2 mm diameter micro tip was immersed at the center level of distilled water to treat the peanut husk powder for 30 min. The sample was treated at adjusted amplitude percentage level of 60 which equivalent to power level of 3.5 W. For indirect ultrasound pre-treatment, 1 g of peanut husk powder was immersed in 250 mL beaker with 200 mL distilled water. The beaker was then placed in the ultrasonic bath (53 kHz-350 W, sk7219HP, KUDOS, China) with tank dimensions of 35 cm × 32 cm × 30 cm for 30 min. The sample was treated at adjusted percentage power level of 100 which equivalent to power level of 3.5 W. Both ultrasound pre-treated peanut husk powder was dried in oven for 24 h and stored in a sample bottle for further use.

2. 4. Batch Adsorption Experiment Batch adsorption experiment was conducted in a 250 mL beaker with 50 mL of heavy metal solution at different initial concentrations (10, 20, 30, 40 and 50 mg/L) at pH 6. 0.2 g of adsorbent was immersed in 250 mL beaker and stirred using stirring plate (Cole- Parner Stable Temp, United State of America) at 350 rpm (conventional method) for 3 h until equilibrium was reached. The concentration of heavy metal was continuously measured at every 0.5 h. Adsorbent was separated from heavy metal solution using centrifugation (SIGMA 2-6E, United Kingdom). Final concentration of heavy metal was measured by using Atomic Adsorption Spectrometer (AAnalyst 400, PerkinElmer, USA). For ultrasound simultaneous adsorption, sonication was applied along with adsorption process. 0.2 g of adsorbent immersed in 250 mL beaker with 50 mL of heavy metal solution was placed in ultrasonic bath with power level of 3.5 W. The equilibrium adsorption capacity (*qe* (mg/g)) was calculated by using following equation:

$$qe = \frac{(Co - Ce)V}{W} \quad (1)$$

where *q*e is the equilibrium adsorption capacity (mg/g), *Co* is the initial concentration of heavy metal (mg/L), *Ce* is the concentration of heavy metal at equilibrium (mg/L), *V* is the volume of the solution (mL) and *W* is the mass of dry sorbent used (g).

2. 5. Effect of pH 0.2 g adsorbent was immersed in 50 mL of 30 mg/L heavy metal solution in a 250 mL beaker at different pH (2 - 8) and stirred using stirring plate at 350 rpm for 3 h. The pH of heavy metal solution was adjusted using 0.1N of NaOH and HCl. Adsorbent was separated from heavy metal solution using centrifugation. Final concentration of heavy metal was measured by Atomic Adsorption Spectrometer. Percentage of heavy metal removal was calculated by the following equation:

$$R(\%) = \frac{(Co-Ce)}{C0} \times 100\% \quad (2)$$

where *R* is the heavy metal removal percentage (%), *Co* is the concentration of heavy metal at initial (mg/L) and *Ce* is the concentration of heavy metal at equilibrium (mg/L).

2. 6. Effect of Dosage Different amount of adsorbent (0.1 - 0.3 g) were immersed in 50 mL of 30 mg/L heavy metal solution in a 250 mL beaker at pH 6. The beaker was placed on stirring plate and stirred at 350 rpm for 3 h. Adsorbent was separated from heavy metal solution using centrifugation. Final concentration of heavy metal was measured by Atomic Adsorption Spectrometer. Percentage of heavy metal removal was calculated by Equation (2).

2. 7. Characterization of Peanut Husk Powder Untreated peanut husk powder and both ultrasound pre-treated peanut husk powder were coated with 600 nm gold layer before scanning the surface features and morphology. The surface features and morphological characteristic of peanut husk powder surface are analyzed by scanning electronic microscopy (JSM-6400, Japan). Surface features and morphology can be observed from the SEM photographs at 1000 magnifications.

3. RESULTS AND DISCUSSION

3. 1. Characterization of Peanut Husk Powder Figure 1 shows the surface features and morphology of (a) untreated peanut husk powder, (b) direct ultrasound pre-treated peanut husk powder and (c) indirect ultrasound pre-treated peanut husk powder which studied by SEM. The untreated peanut husk powder in Figure 1 (a) indicated a smooth surface with less pores structure on it before ultrasound pre-treatment. Figure 1 (b) and (c) show the formation of new pores and rough surface after ultrasound pre-treatment. Indirect ultrasound cause the more deep pores formation on the powder surface compared with direct ultrasound.

The pores formation are due to ultrasound cavitation effect. The violent collapse of bubbles during cavitation caused high shearing effect and strong acoustic steam in the medium [24]. It generates a microjet to hit the solid surface and lead to pore formation on the surface [25]. It is suggested that the higher temperature of water medium during indirect ultrasound treatment (45℃) has lower liquid tensile strength than direct ultrasound treatment (32℃). This caused bubbles cavitation formed more easily and collapsed more violently at low tensile strength of liquid; thus created higher number of deep pores at the powder surface [26].

3. 2. Effect of Different Initial Concentration and contact time The result shows that initial concentration of copper (II) ion is important in adsorption process. This is because concentration provides driving force to overcome mass transfer resistance of heavy metal ion and solid phase [27].

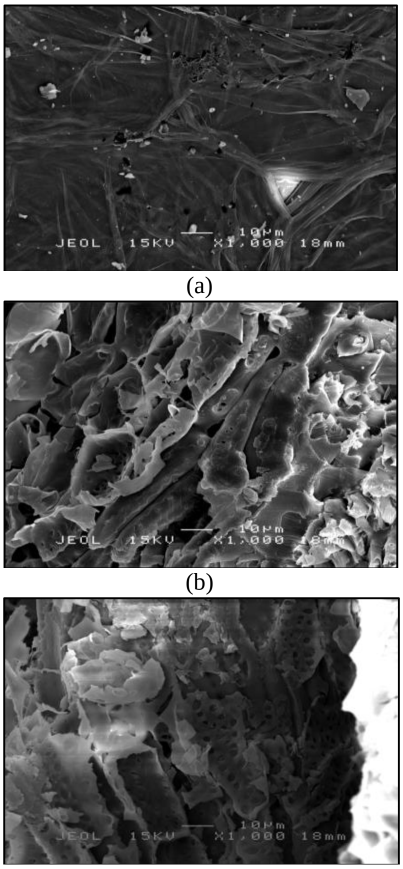

Figure 1. Image of scanning electron microscopy for (a) untreated, (b) direct ultrasound and (c) indirect ultrasound pre-treated peanut husk powder 3.5 W

Figure 2 shows the effect of different initial concentrations (10 – 50 mg/L) and contact times (0.5 – 5 h) on the removal of copper (II) ion by untreated and ultrasound pre-treated peanut husk powder, and ultrasound simultaneous adsorption process.

It showed that the rate of copper (II) ion removal was rapid at the first 0.5 h, it then slowed down after 0.5 h and reached adsorption equilibrium. This is probably due to high availability of adsorption site or pores on the surface of peanut husk powder at the beginning [28]. The removal rate of copper (II) ion was slowed down after 0.5 h because the adsorption site or pores were gradually occupied.

Untreated peanut husk powder need 1.5 and 3 h to reach adsorption equilibrium at 10 - 20 mg/L and 30 - 50 mg/L copper (II) ions, respectively. At high concentration, peanut husk powder take longer time to reach adsorption equilibrium because the solution contain high amount of copper (II) ions. The equilibrium time of indirect ultrasound pre-treated peanut powder has shortened 0.5 h at 10 - 20 mg/L concentration of copper (II) ions. The adsorption capacity of direct ultrasound pre-treated peanut husk powder has increased from 1.86 to 10.68 mg/g with the increases of heavy metal initial concentration from 10 to 50 mg/L. The percentage removal of copper (II) ions for indirect ultrasound pre-treated peanut husk powder has overall increased about 2% compare to the direct ultrasound pre-treated peanut husk at copper (II) initial concentration of 10 to 50 mg/L. It is obeserved that solution with higher initial concentration of copper (II) has higher percentage removal. The overall percentage removal of copper (II) by indirect ultrasound pre-treated peanut husk powder was about 22% higher than simultaneous ultrasound and untreated peanut husk powder at copper (II) initial concentration of 50 mg/L. These results showed that indirect ultrasound pre-treated peanut husk powder is more efficient to remove the copper (II) ions from the solution.

The adsorption rate of copper (II) ion by direct and indirect ultrasound pre-treated peanut husk powder was higher than untreated and ultrasound simultaneous adsorption. It is suggested that ultrasound waves induced bubbles cavitation surrounding the solid surface of husk powder, which reduced the mass transfer boundary layer to ease the adsorption process and creating more porous structure on the surface at the adsorption site [29]. Therefore, ultrasound pre-treated husk powder can enhance the mass transfer rate of copper (II) ions at the powder surface to reach adsorption equilibrium quickly and increasing the adsorption capacity [30].

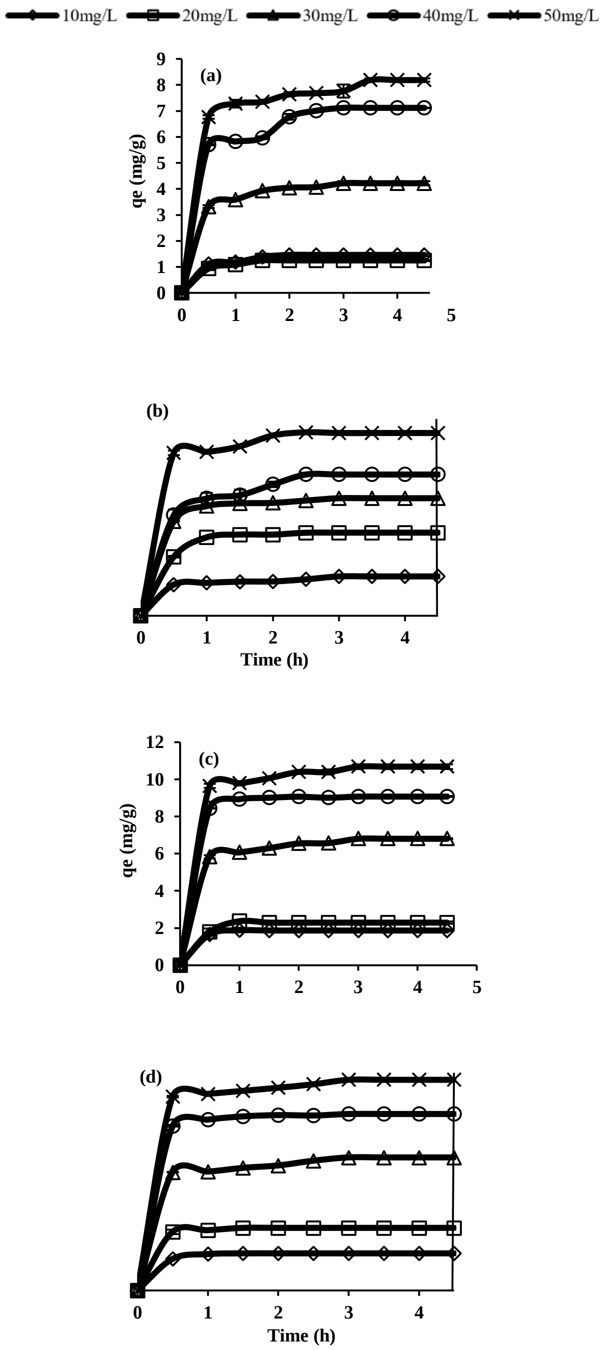

Figure 2. Effect of different initial concentration of copper (II) ion and contact time on heavy metal removal by (a) untreated peanut husk powder, (b) ultrasound simultaneous adsorption, (c) direct and (d) indirect ultrasound pre-treated peanut husk powder (Temperature = 30℃, adsorbent dose = 0.2 g, pH 6, heavy metal concentration = 10-50 mg/L, contact time = 0.5-3 h, stirring speed = 350 rpm).

3. 3 Effect of pH Solution pH plays an important role in removal of heavy metal. It was significantly affected the percentage of copper (II) ion removal. Experiment was conducted with 30 mg/L copper (II) ions at different pH values (2-8) with 3 h contact time. Figure 3 shows that solution condition at pH 2 gave the lowest percentage removal of copper (II) ions. Percentage removal of copper (II) ion was gradually increased from pH 2 to 6 and decreased at pH 8. The maximum percentage removal of copper (II) ion was achieved at pH 6. Low percentage removal of copper (II) ion at acidic solution is due to higher number of hydrogen cation competing with copper (II) cation for the vacant sites at peanut husk powder [31]. High percentage removal of copper (II) ion at increasing solution pH is because negatively charge attached more on peanut husk powder surface which may increase the electrostatic attraction between copper (II) cation and peanut husk powder [32]. The removal of copper (II) ion was decreased at pH 8 due to metal hydroxide formation [33].

It was observed that percentage removal of copper (II) ion was increased with ultrasound pre-treatment. Indirect ultrasound pre-treated peanut husk powder has achieved the highest percentage removal of copper (II) ion with 92.07 % at pH 6. Indirect ultrasound pre-treated peanut husk powder shown better performance compared to direct peanut husk powder. At pH 6, indirect ultrasound pre-treated powder has higher percentage removal on copper (II) ion compared to untreated peanut husk powder and ultrasound simultaneous adsorption at 57.07% and 19.63%, respectively. Temperature of distilled water has increased from 27.5 ℃ to 32 ℃ and 45 ℃, respectively during direct and indirect ultrasound treatment of peanut husk powder at power level 3.5 W. Bubbles cavitation are more easily occured at high temperature due to low liquid tensile stress and those cavitation energy caused more pores formation on peanut husk powder surface [26].

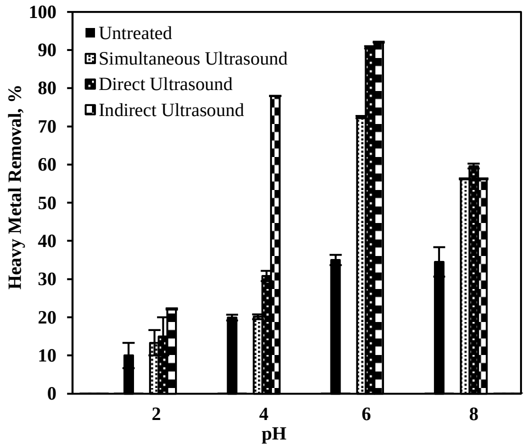

Figure 3. Effect of solution pH on copper (II) ion adsorption by untreated and ultrasound pre-treatment peanut husk powder, and simultaneous ultrasound (Temperature = 30 ℃, adsorbent doses = 0.2 g, pH 2-8, dye initial concentration = 30 mg/L, contact time = 3 h, stirring speed = 350 rpm).

Thus, generate more active site with negative charge on peanut husk powder surface and lead to more copper (II) cation attached on its surface.

3. 4. Effect of Dosage Adsorbent dosage is an important factor because it determine the adsorption capacity of an adsorbent at different initial concentration of the adsorbate [35]. The dosage effect of peanut husk powder was studied in the range from 0.1 to 0.3 g. Figure 4 shows that the percentage removal of copper (II) ion was increasing with dosage of peanut husk powder. This is because high dosage of peanut husk powder has increased the surface area and adsorption site for copper (II) ion to bind with it [34]. The percentage removal of copper (II) ion by 0.1 g indirect ultrasound pre-treated peanut husk powder was 23.20 % higher than 0.3 g untreated peanut husk powder. The high adsorption efficiency given by ultrasound is suggested due to bubbles cavitation effects, which increase the surface area by producing more adsorption sites of pore on the peanut husk powder surface. Hence, with the application of ultrasound pre-treatment, low dosage of peanut husk powder was able to attains high percentage removal of copper (II) ion.

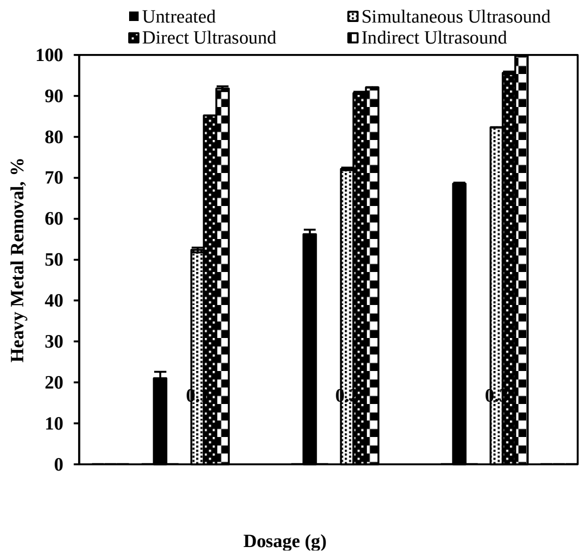

Figure 4. Effect of dosage of peanut husk powder on copper (II) ion adsorption by untreated and ultrasound pre-treatment peanut husk powder, and simultaneous ultrasound (Temperature = 30 °C, adsorbent doses = 0.1–0.3 g, pH 6, copper (II) concentration = 30 mg/L, contact time = 3 h, stirring speed = 350 rpm)

4. CONCLUSION

Both ultrasound pre-treated peanut husk powders have higher copper (II) ion removal than untreated peanut husk powder and ultrasound simultaneous adsorption.

Ultrasound pre-treated peanut husk powder has increased the rate of adsorption process, and shorten the equilibrium time to achieve higher percentage removal of copper (II) ion. The highest percentage removal of copper (II) ion was 92.07% at pH 6 by indirect ultrasound pre-treated peanut husk powder. Higher dosage of peanut husk powder caused more copper (II) ion removal. With the cavitation effect induced by ultrasound, 0.3 g of indirect ultrasound pre-treated peanut husk powder was able to achieve the highest copper (II) ion removal of 99.79%. Ultrasound pre-treated peanut husk powder is more practicable to apply as a ready adsorbent for copper (II) ion in industry compared to ultrasound simultaneous application. This study showed that small amount of ultrasound pre-treated peanut husk powder which was able to remove higher quantity of copper (II) ion compared to high dosage of untreated powder.

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Peanut Husk Powder

چکیده

جداسازی یون مس (II) از طریق جذب سطحی بر روی پودر پوسته بادام زمینی را می توان با پیش فراوری به روش پیش تیمار فراصوت افزایش داد. ساختار متخلل و سطح تماس پودر پوسته بادام زمینی از طریق پیش تیمار مستقیم (میله فراصوت) و پیش تیمار غیر مستقیم (حمام فراصوت) بهبود داده می شود. در مطالعات قبلی، محققان استفاده همزمان فرآیند جذب سطحی و پیش تیمار فراصوت را به کار برده اند. هرچند، این روش برای فراوری مقادیر زیاد فلزات سنگین در فاضلاب مناسب نمی باشد. در تحقیق پیش رو، جداسازی یون مس (II) از طریق جذب سطحی با استفاده از پودر پوسته بادام زمینی پیش فراوری شده به روش پیش تیمار فراصوت مستقیم و غیر مستقیم با پودر پوست بادام زمینی فراوری نشده به عنوان جاذب و فرایند جذب سطحی و پیش تیمار فراصوت همزمان مقایسه شده است. مشخصات پودر پوست بادام زمینی به کمک میکروسکوپ الکترونی (SEM) تعیین گردیده است. در این تحقیق تاثیر متغییرهایی نظیر غلظت اولیه یون مس (II) (10-50 میلی گرم/لیتر)، زمان تماس (0.5–3 ساعت)، pH (2–8) و دوز جاذب (0.1–0.3 گرم) بر روی فرآیند جذب سطحی ارزیابی گردیده است. نتایج حاکی از آن است که 3 ساعت زمان تماس برای جذب تعادلی یون مس بر روی پودر پوست بادام مورد نیاز بوده است. همچنین بالاترین میزان جذب یون مس (II) به مقدار 99.79 درصد در pH 6 و دوز 0.3 گرم با استفاده از پودر پوست بادام زمینی پیش فراوری شده به روش پیش تیمار فراصوت غیر مستقیم به دست آمده است. این مقدار به ترتیب 57.07 درصد و 19.63 درصد بیشتر از جاذب فراوردی نشده و فرایند همزمان جذب و پیش تیمار بوده است. هر دو جاذب پیش فرآوری شده به روش آوادهی فراصوت بهبود قابل توجهی برای جذب یون مس (II) نسبت به جاذب فراوری نشده و فرآیند جذب سطحی و پیش تیمار فراصوت همزمان نشان داده اند.

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