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EFFECTS OF OSMOTIC DEHYDRATED AND ULTRASOUND PRE-TREATED ORANGE PEEL ON DYE REMOVAL

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ABSTRACT

In this study, the adsorption performance of orange peel with osmotic dehydration (OD) and ultrasound (US) were studied. The efficiency of the osmotic dehydration pre-treated orange peel at different sucrose concentration (20 – 60 °Brix) in dye adsorption with several conditions of initial dye concentration (20 – 60 mg/L) and dosage (0.05 – 0.15g) was studied and compared with untreated adsorbent. The optimum concentration of the hypertonic solution was 20 °Brix (OD-20) which gave the higher adsorption efficiency compared with the control and other concentration of hypertonic solution. The adsorption efficiency of OD-20+US pre-treated orange peel on 20 mg/L dye concentration with 150 mg of dosage was 6.14% and 19.32% higher than OD-20 and untreated orange peel respectively. Furthermore, OD-20+US also increases the desorption efficiency by 5.81% and 13.37% when compared with OD-20 and untreated orange peel respectively. With OD-20, the number of active pore availability increases due removal of moisture content by sucrose particles and US able to create microscopic channels in porous materials to enhance the moisture removal and captures of dye ions.

Contribution/Originality: The paper's primary contribution is finding that combination of ultrasound and osmotic dehydration treatments has positive impact on the preparation of adsorbent. It is able to generate the adsorbent with high adsorption capacity and reusability by introducing the treatments.

1. INTRODUCTION

In this century, dyes are commonly used by many industries to colour and enhance the appearance of their product. These industries include food, paper, plastics, paint and also textile industries. The usage of dyes will eventually generate a considerable amount of colored wastewater due to less efficient in dyeing techniques and it is required to be treated before discharging it. The application of reactive dyes in a process will cause more than 50% of dyes lost directly into the water streams [1]. Colour dyes are greatly visible, easily detectable and unpleasant in water, even though the concentration is extremely low [2]. Moreover, the application of dye in industrial processes will produce colored water which may slow down the photosynthesis process. This situation will eventually affect the environment and aquatic ecosystems. Major problem to the environment may occur if they are broken down anaerobically in the sediment [3].

In the world-wide, there are approximately 800,000 tons of dyes produced annually and there are more than 100,000 types of dyes available in the market [4]. The most common pollutant found in industrial wastewater is the synthetic dyes due to its good solubility in water. Many of these dyes are toxic and harmful to the environment,

especially towards aquatic life. Thus, the removal of colour dyes from wastewater is a very important process before discharging it to the environment. However, it is very hard to treat wastewater which contains dyes in it as they are recalcitrant organic molecules which resists to aerobic digestion and have good stability under heat condition and presence of oxidizing agent [5]. In present, colour dyes can be removed through biological, chemical and physical treatment. Biodegradation methods such as fungal decolorization and microbial degradation are the most common and economical biological method to remove dye from water [6]. Chemical treatment involves coagulation, flocculation, floatation and filtration process [7]. Physical treatment involves membrane filtration process and also adsorption techniques [7]. In physical treatment, adsorption is widely used in dye removal process due to insensitivity towards toxic pollutants, ease of operation and highly effective compared to other treatment [8]. Adsorption technique uses solid adsorbents to eliminate a definite range of toxic and harmful pollutants from wastewaters. Conventional adsorbents which commonly applied by industries to remove dyes include activated carbon, zeolites, clays, chitin and chitosan [9]. Among all types of solid adsorbents, activated carbon is the most favored one due to high adsorption performance. However, activated carbon is very expensive in term of regeneration and operation [10]. Therefore, many researchers are looking into the replacement of high cost activated carbon with alternative adsorbents.

In present days, many raw agricultural solid wastes such as corn cob [11] rice husk [12] barley husk [13] orange peel [14] sawdust [15] and wheat straw [2] have been used as adsorbent to eliminate dye from water by many industries. They are commonly used as adsorbent due to high availability in large quantities, unique physicochemical characteristics and low cost. Before using it to remove dyes from wastewater, agricultural wastes have to undergo drying process to ensure the structure of the solid waste is free from water and have the room to trap colour dye. The drying process is very important in adsorbent preparation. Ultrasound and osmotic dehydration had been widely applied in drying process as to enhance the moisture removal and shorten the drying time. In this study, both ultrasound and osmotic dehydration treatments were used to prepare the adsorbent to further apply in adsorption study. It is suggested that ultrasound can improve the moisture removal from the sample with the assistance of osmotic dehydration by creating microscopic channels in the sample cell structure whilst it contributed as the adsorption sites in the adsorption process later for dye particle to bind with [16].

The first part of this study was to determine the drying kinetic of the osmotic dehydration pre-treated orange peels with different sucrose concentration (°Brix). Then in the second part of the study, the osmotic dehydration pre-treated orange peels were used in the adsorption study at different dye concentration and with different adsorbent dosages. The effect of osmotic dehydration treatment on the orange peel at optimum sucrose concentration in drying and dye adsorption performances was further compared with the assistance of ultrasound.

2. MATERIALS AND METHODS

2.1. Sample Preparation

Orange was purchased from local market in Cheras, Kuala Lumpur, Malaysia. The orange peel was separated from the pulp by using hand and rinsed with distilled water to remove the dirt from the surface of the orange peel. It was cut into slabs with equivalent size and thickness. The dimension of the slab was approximately 10 mm x 10 mm x 3 mm. All the samples were prepared on the same day to minimize the difference of moisture content between the samples. Sample was wrapped with plastic film to prevent water loss.

2.2. Osmotic Dehydration Pre-Treated Adsorbent

The hypertonic solutions of 20, 40 and 60 °Brix were prepared by dissolving 20, 40 and 60 g of sucrose respectively in 100 g of distilled water. The sucrose and water was evenly mixed with a glass rod to ensure the sucrose is completely dissolved in water. After that, 15 g of orange peels were weighted and immersed in the hypertonic solution of different concentrations (20, 40 and 60 °Brix) for an hour. The sample was then removed

from the solution and dried in an oven (AE-18N, Pensonic, Malaysia) at 100 °C. The weight of the sample was measured by using a weighing balance at every 5 min until a constant weight was obtained. The total moisture content percentage of the sample was calculated by using Equation 1 [17]. The dried sample were grinded into powder form by using the grinding machine (RW-5082, Richwell, Malaysia) and stored in an air tight container for further use in the adsorption process.

$$\text{Total moisture content } (\%) = \frac{\text{Weight}_{\text{Wet sample}} - \text{Weight}_{\text{Dried sample}}}{\text{Weight}_{\text{Dried sample}}} \times 100\% \quad (1)$$

2.3. Ultrasound-Assisted Osmotic Dehydration Pre-Treated Adsorbent

15 g of orange peels were immersed into 100 g of hypertonic solution in a 2 L conical flask. The hypertonic solution with optimum concentration which gives the highest adsorption efficiency was used as the sonication medium in the ultrasound treatment. The conical flask was then immersed in the ultrasound bath tank (53 kHz-350 W, sk7210HP, KUDOS, China) and treated at 90% sonication power for an hour at 40 °C. The ultrasound-assisted osmotic dehydration treated sample was then dried in an oven at 100 °C until a constant weight was achieved. The dried sample was grinded into powder form by using the grinding machine (RW-5082, Richwell, Malaysia) and stored in an air tight container for further use in the adsorption process. The ultrasound and osmotic non-treated orange peels were used as control in this study.

2.4. Adsorption Experiment

Batch adsorption study was conducted by adding 100 mg of adsorbent into 100 mL of crystal violet (R&M marketing, Essex, United Kingdom) dye solution at different initial concentrations of 20, 40 and 60 mg/L in a 250 mL beaker. The beaker was placed on a magnetic stirrer (C-MAG HS 7, IKA, Malaysia) and stirred at 700 rpm. The dye concentration was measured at every 10 min by using the UV-Vis spectrophotometer (Hitachi U-2900 Spectrophotometer, Japan) at a wave-length of 590 nm. The experiment was continued until the dye concentration become constant.

The adsorption capacity at time t, q_t (mg/g) and equilibrium adsorption capacity, q_e (mg/g) were calculated by using Equation 2 and Equation 3 respectively. These values were further used to determine the isotherms of kinetics of the adsorption. The total percentage removal of dye by the absorbent was calculated by using Equation 4.

$$q_t = \frac{v(C_o - C_t)}{w} \quad (2)$$

$$q_e = \frac{v(C_o - C_e)}{w} \quad (3)$$

$$\%\ Removal = \frac{C_o - C_e}{C_o} \times 100\% \quad (4)$$

Where C_o (mg/L) represents the initial dye concentration, C_t (mg/L) represents the remaining dye concentration at time t (min), and C_e (mg/L) represents the equilibrium concentration of absorbent after the adsorption. w (g) indicates the adsorbent dosage and v (L) represents the volume of the dye solution. The batch adsorption experiment was then repeated by adding 50 and 150 mg of adsorbents respectively into 100 mL of dye solution with initial concentration of 20 mg/L in a 250 mL beaker and agitated at 700 rpm. The experiment was conducted at room temperature condition.

2.5. Desorption Experiment

The samples of untreated orange peel, orange peel treated with osmotic dehydration at optimum concentration, and orange peel treated with ultrasound-assisted optimum osmotic dehydration were used for desorption experiment. Each sample with dosage of 150 mg were added into a 100 mL dye solution with concentration of 20 mg/L in a 250 mL beaker and agitated for 700 rpm. After adsorption, the adsorbent was removed from the dye solution through centrifuge machine (2-6E, Sigma Laborzentrifugen, Germany) at 3000 rpm for 12 min. The absorbent was added into a 250 mL beaker containing 100 mL of 1 M NaCl and agitated at 700 rpm for 160 minutes. The concentration of the dye was measured every 10 min by using the UV spectrometer. The percentage of desorption was calculated using Equation 5 and compared with the adsorption percentage.

$$\% \, Desorption = \frac{C_d}{C_a} \times 100\% \tag{5}$$

where C_a (mg/L) is the amount of dye adsorbed by the adsorbent and C_d (mg/L) is the amount of dye in the solution at respective time t (min) [18].

2.6. Characterization of Orange Peel Powder

Scanning electron microscopy (SEM) viewing was conducted to analyze the surface structure of the orange peel before and after osmotic dehydration treatment at optimum concentration, with and without ultrasound pre-treatment process. A small amount of each sample was attached on a metal plate and gold coated with a sputter coater (BALTEC SCD 005, ZEQ-M Trading & Service, Malaysia). After coating, the sample was scanned with scanning electron microscopy (JSM-IT100, JEOL, Malaysia). The image of the sample was magnified in a variety of magnification range to observe the surface structure of the sample in details.

3. RESULTS AND DISCUSSION

3.1. Effect of Osmotic Dehydration and Ultrasound on Drying

Moisture content in the orange peel is very important to be removed for its adsorbent preparation. Osmotic dehydration and ultrasound pre-treatment will eventually affect the drying time of a sample [19]. The researcher proved that both pre-treatments will increase the diffusivity of water from the sample during the air drying process. The osmotic pre-treated sample will result in water loss and sugar gain due to diffusivity of sugar particle in the hypertonic solution [19]. Furthermore, ultrasound treatment which creates the microscopic channel allowed the diffusion of water to be much easier [16]. Figure 1(a) shows the total moisture content percentage of sample treated with osmotic dehydration at different concentration of hypertonic solution, i.e. 20 °Brix (OD-20), 40 °Brix (OD-40) and 60 °Brix (OD-60). The results were compared with the sample without treatment and sample treated with ultrasound-assisted osmotic dehydration at 20 °Brix at 90% sonication power (OD-20+US).

The results show that orange peel with pre-treatment has a lower total moisture content compared with the untreated orange peel. In OD treatment, it shows that the moisture content remained inside the adsorbent with OD-60 is much lesser compared to OD-40 and OD-20. It shows that as the sucrose concentration in hypertonic solution increased, the moisture content remained in the orange peel after treatment will be lower. This might due to the fact that the water is forced out from the sample by the sugar particle. Therefore, when the concentration in hypertonic solution increases, the sugar particle forcing into the sample will also increase and lead to higher percentage of water being forced out from the sample. At lower concentration, the sugar particle diffusing into the sample is lower and hence the water remained inside the sample is much higher. Moreover, the percentage of total moisture content inside the sample with OD-20 has reduced 5% with the assistance of ultrasound. The reduction in moisture content with assistance of ultrasound might due to the creation of microscopic channels in the internal

structure of the orange peel. With the development of microscopic channels, more sugar particle from the hypertonic solution will able to diffuse in and force out the water from the orange peel. Figure 1(b) shows that the drying time for OD-60 is only around 120 minutes while the drying time for untreated sample, OD-20 and OD-40 is above 140 minutes. Although the moisture content of sample OD-20+US is higher compared to OD-40 and OD-60, however, its drying time is much shorter compared with other samples, where it only took around 100 minutes to dry completely. This is suggested that the microscopic channels created by ultrasound have increased the water diffusion during the drying process.

3.2. Effect of Dye Concentration on Adsorption

In the OD treatment without ultrasound for the adsorption of 40 mg/L dye solution, OD-20 has the highest equilibrium adsorption capacity, followed by OD-40 and OD-60 as showed in Figure 2(a). This might due to lower sugar component remained in the cell structure causing higher amount of pores available to trap the dye ions. The result further shows that when OD-20 was assisted with ultrasound treatment, its adsorption capacity was 8.06% higher than the OD-20 sample without ultrasound treatment. It is suggested that the porous structure and microscopic channel which created by ultrasound cavitation effect on the sample have contributed as the adsorption sites to bind with the dye ions [20, 21]. The adsorption capacity of all the adsorbents with different pre-treatment show a similar trend at all the dye concentrations from 20 mg/L to 60 mg/L. As the concentration increased, the adsorption capacity will also increase.

Figure 2(b) shows the percentage of dye removal for all the adsorbents at different level of dye concentrations. As the concentration of dye solution increased, the percentage of dye removal by the adsorbent reduced. The dye removal percentage of untreated orange peel at 20 mg/L dye concentration shows 23% and 38% higher than 40 mg/L and 60 mg/L dye concentration respectively. The percentage of dye removal for orange peel with OD treatment is always higher compared with untreated orange peel at all the dye concentrations. It is observed that OD-20 has the highest percentage in dye removal compared to other OD treatment with different brix level. It shows about 23 – 30% higher than the control at dye concentration between 20 – 60 mg/L. Moreover, the percentage of dye removal of OD-20 has further increased about 10 – 13% for dye concentrations between 20 – 60 mg/L with the assistance of ultrasound.

3.3. Effect of Adsorbent Dosage on Adsorption

Figure 3(a) shows the adsorption capacity of adsorbent with different pre-treatments in 40 mg/L of dye solution decreased when the adsorbent dosage increased from 50 mg to 150 mg. When the dosage of adsorbent increases, the number of active sites on the adsorbent structure will also increase, thus increases the opportunity for the dye particles to bind with the adsorbent, hence lead to higher percentage of dye removal as showed in Figure 3(b) [22]. However, the adsorption capacity of the adsorbent during the equilibrium is depends on the ratio between the active site to the concentration of dye in the solution. Lower dosage of adsorbent shows higher adsorption capacity at the end of the adsorption process as most of the active sites are filled with dye particles after the adsorption, while higher dosage of adsorbent shows lower adsorption capacity as the ratio of active sites availability in the structure is higher than the amount of dye particles to be adsorbed in the solution. Higher dosage of adsorbent gave dramatic positive impact on decolorization and shows a direct proportional relationship between the adsorbent dosage and the percentage of dye removal [23]. The adsorbent pre-treated with lowest brix of hypertonic solution gave the highest percentage of dye removal as showed in Figure 3(b). At the highest dosage of 150 mg, OD-20 pre-treated orange peel indicated the highest increased about 37% compared to the control. Whilst with the assistance of ultrasound, OD-20+US pre-treated orange peel had further increased the dye removal percentage about 49% compared to the control.

3.4. Desorption Study

The desorption study on adsorbent was carried out after the adsorption process to identify the potential of reusability of the particular adsorbent. The reusability of an adsorbent is important in term of costs and industrial application. This study is able to outline the nature of dye adsorption process and the recovery of crystal violet from adsorbent with different pre-treatment. Before conducting the desorption process, adsorption of crystal violet with 20 mg/L on 150 mg of adsorbents (control, OD-20 and OD-20+US) were carried out. During the adsorption process, the non-treated adsorbent (control), OD-20 and OD-20+US has adsorbed 54.30%, 67.48% and 73.62% of dye ions respectively as shown in Figure 4. In the desorption process, 1 M of NaCl was used and the dye was desorbed for 160 min. During the desorption process, the control, OD-20 and OD-20+US adsorbents manage to desorb 78.05%, 85.61% and 91.42% of dye ions respectively from the total amount of dye absorbed during the adsorption process. The adsorbent showed higher adsorption and desorption percentage with the assistance of ultrasound during the osmotic dehydration treatment. Therefore, it can be concluded that the adsorbent with osmotic dehydration and ultrasound pre-treatments will have a better potential to be reused.

3.5. Scanning Electron Microscopy (SEM)

The surface feature of the untreated adsorbent, and adsorbents pre-treated with OD-20 and OD-20+US were identified by using the scanning electron microscopy. Figure 5 shows the morphology and surface feature of the untreated and OD-20 pre-treated adsorbents are with magnification of 300, while OD-20+US pre-treated adsorbent with magnification of 450. Through the observation on Figure 5(a), the non-treated adsorbent has the potential on adsorption due to the availability of the pores structure naturally on its sample structure. For OD-20 pre-treated adsorbent, the number of pores appeared in the structure is higher compared with untreated orange peel and it also more evenly distributed on the structure.

When orange peel is treated with osmotic dehydration, the hypertonic solution will actually break the cell wall and enlarge the pore size and the pore depth [24]. At the same time, the sugar content from the hypertonic solution will actually diffuse into the structure and force part of the water to diffuse out from the structure [25]. The larger pore size caused by OD will give a better adsorption capacity compared with the untreated adsorbent. With the assistance of ultrasound, it shows that the pore size on the structure is slightly bigger and the depth is deeper compared to the adsorbent which only undergo OD treatment. Ultrasound will eventually create microscopy channels in the structure causing more water to be removed from the structure and enhance the drying effect. When the structure is larger, it causing more sugar particle which trapped inside the structure during osmotic dehydration to escape from the structure through diffusion. This eventually increases the active pore surface area, which allows more dye ions to be adsorbed on the active site of the adsorbent.

4. CONCLUSIONS

In conclusion, as the concentration of the hypertonic solution increased, the moisture content remained inside the orange peel decreased. However, a lower adsorption capacity was observed when the concentration of hypertonic solution increased due to sucrose content that diffused into the orange peel was increased and reduced the active pore surface area for adsorption. Therefore, orange peel pre-treated with OD-20 has the highest adsorption efficiency compared to OD-40 and OD-60. Furthermore, the application of ultrasound on osmotic dehydration was able to reduce the moisture contents and improve the adsorption capacity of the adsorbent on crystal violet. The research has proven that the adsorbent pre-treated with osmotic dehydration and ultrasound has high potential to be reuse with its high desorption capacity on dye.

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Competing Interests: The authors declare that they have no competing interests.
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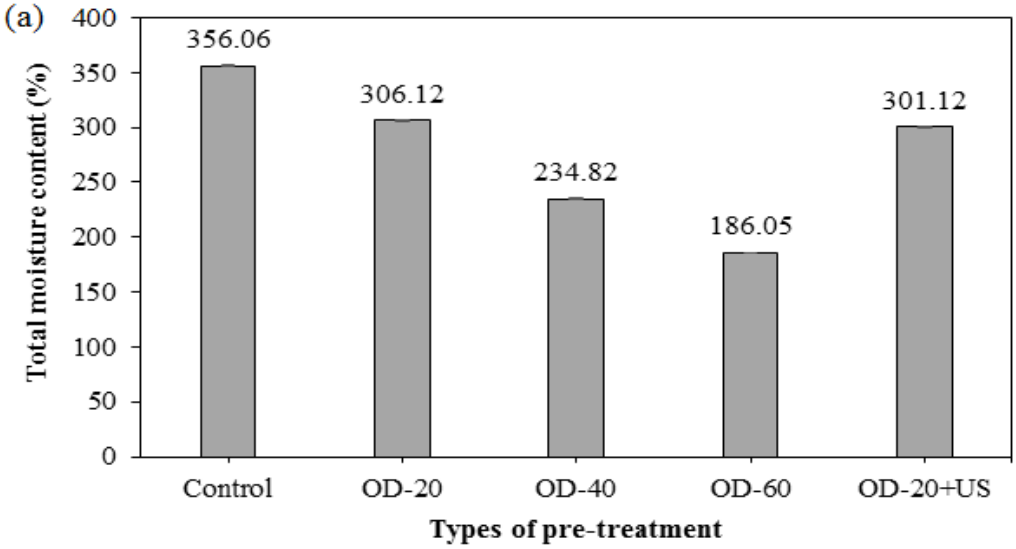
FIGURE CAPTIONS



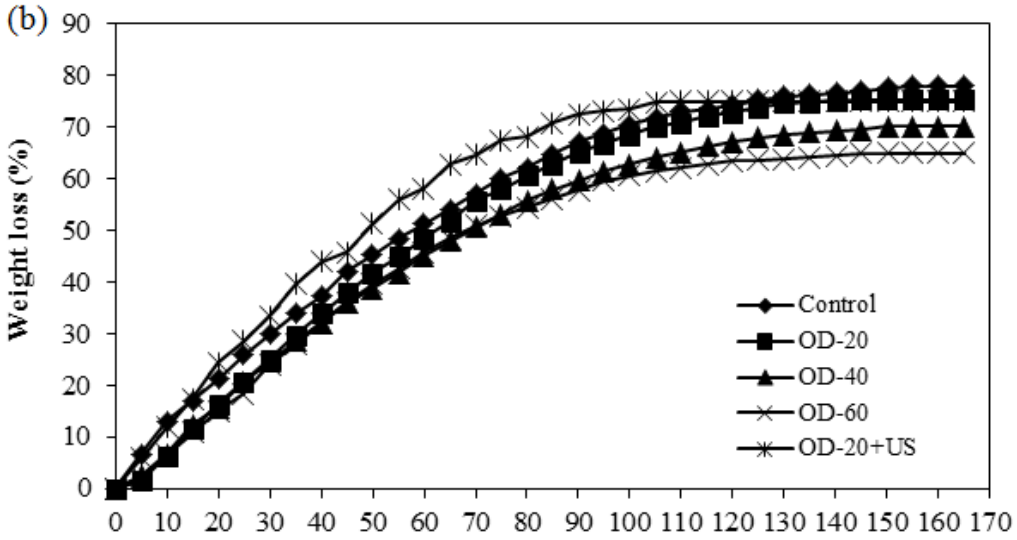


Figure-1. Percentage of (a) total moisture content and (b) weight loss of orange peel after osmotic dehydration (OD) and ultrasound (US) pre-treatments.
Source: The data was obtained from experimental works.

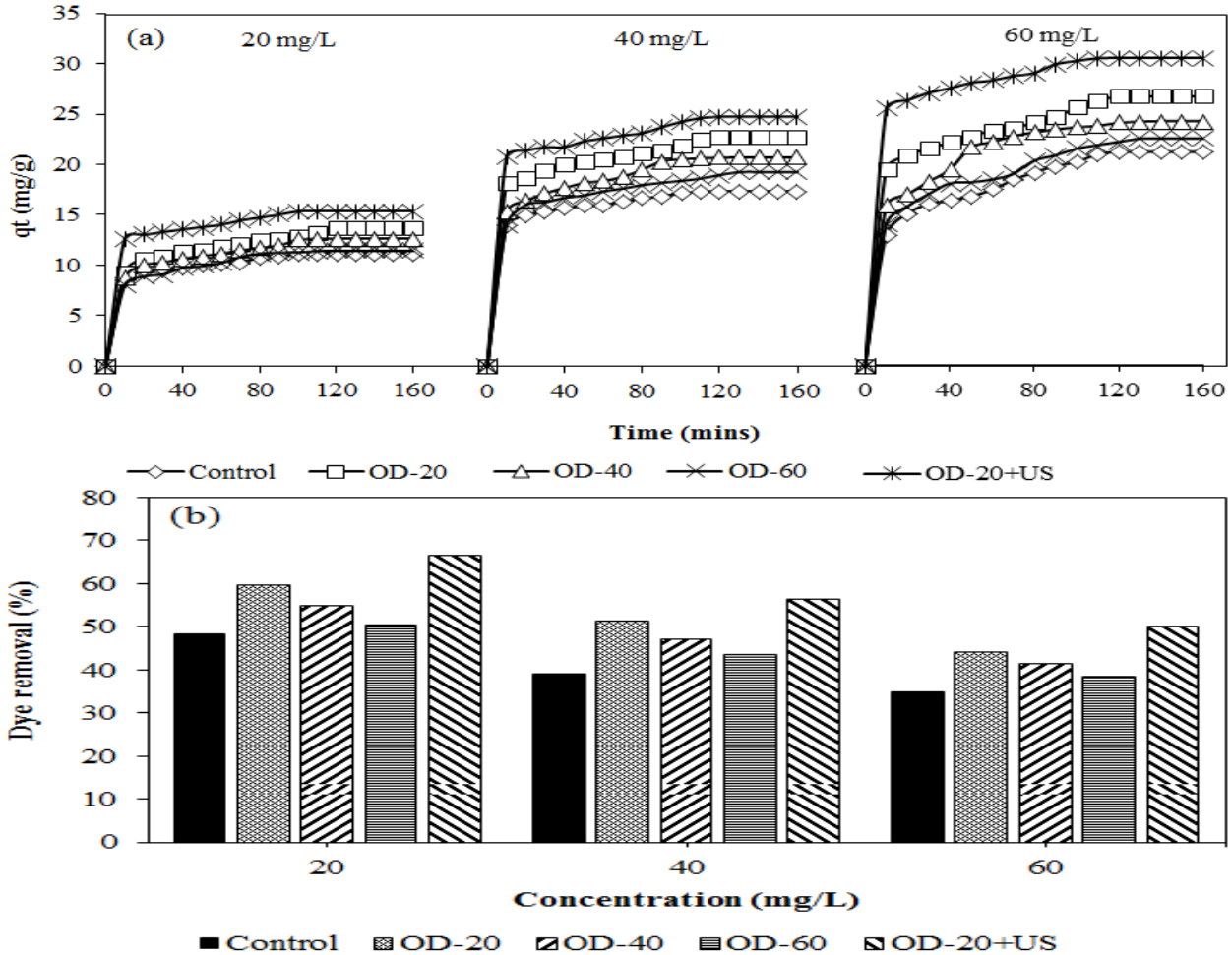


Figure-2. (a) Adsorption capacity and (b) percentage of dye removal for adsorbent with different pre-treatment at dye concentration of 20 mg/L, 40 mg/L and 60 mg/L.
Source: The data was obtained from experimental works.

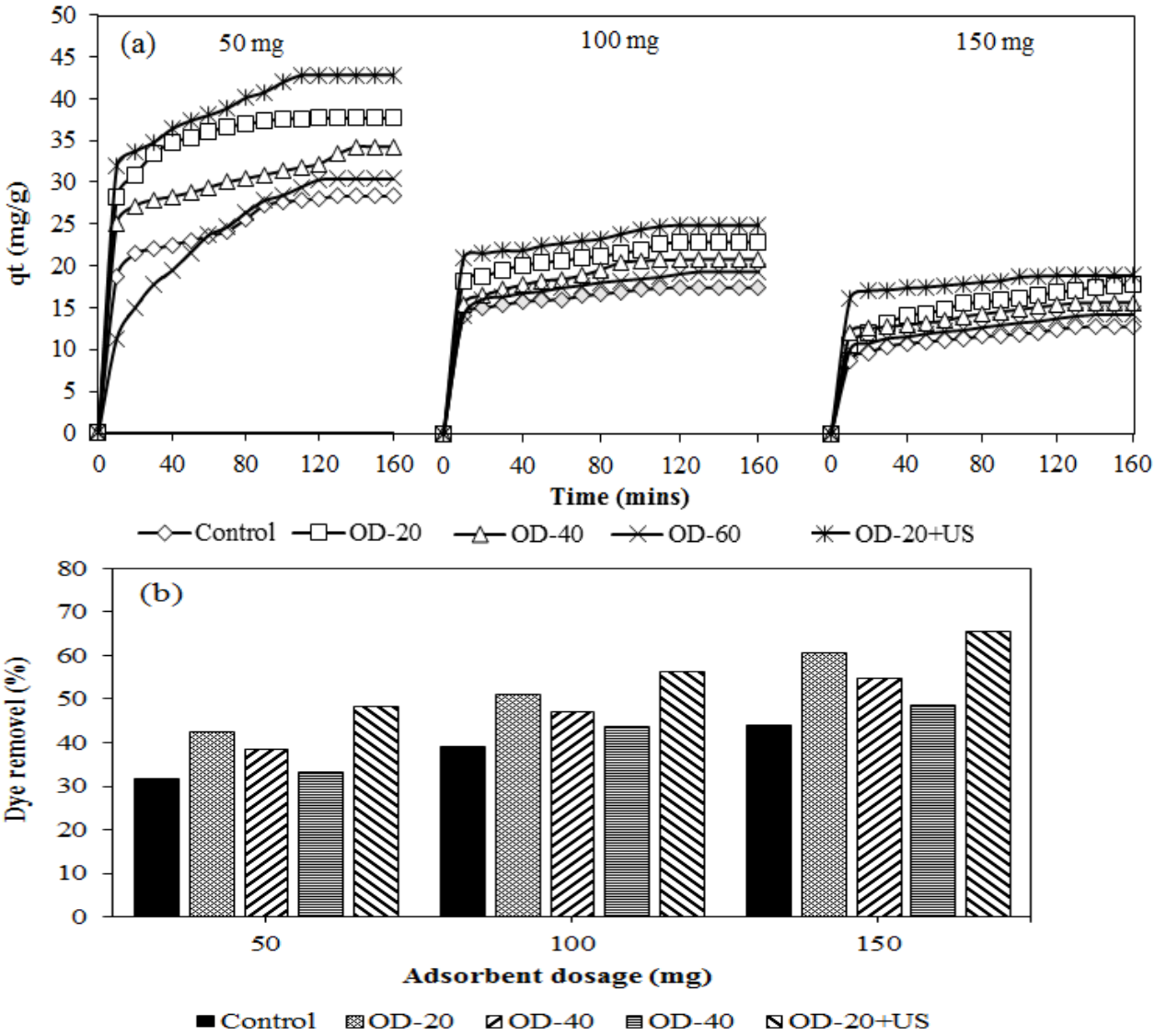


Figure-3. (a) Adsorption capacity and (b) percentage of dye removal for adsorbent with different pre-treatment at adsorbent dosage of 50, 100 and 150 mg.
Source: The data was obtained from experimental works.

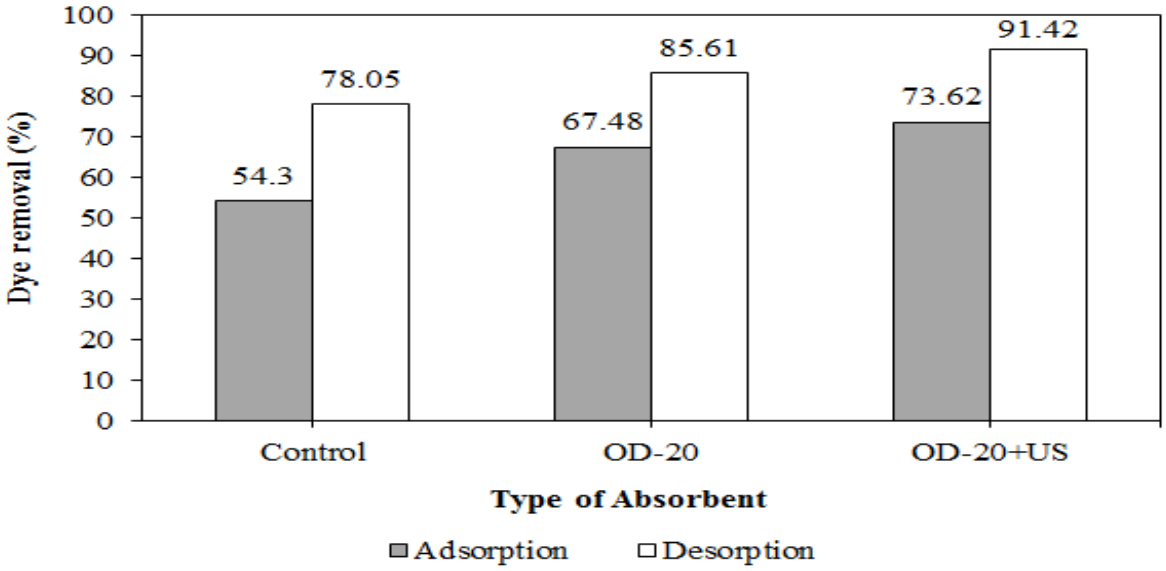


Figure-4. Percentage removal of dye during adsorption and desorption.
Source: The data was obtained from experimental works.

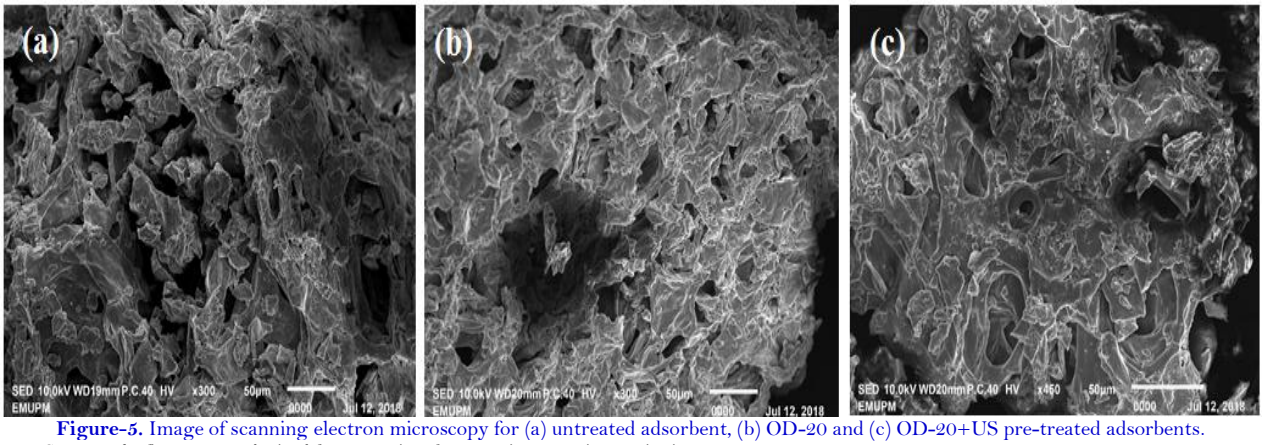


Figure-5. Image of scanning electron microscopy for (a) untreated adsorbent, (b) OD-20 and (c) OD-20+US pre-treated adsorbents.
Source: The figures were obtained from scanning electron microscopy image viewing.