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Utilization of Magnetic Biochar from Palm Shell as An Adsorbent for Removal of COD, Total Suspended Solid, Oil and Grease in Greywater

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Abstract: Greywater is wastewater from domestic activities sourced from kitchens, bathrooms, and laundry water. Greywater discharged directly into the environment can cause a decrease in surface water quality due to its high organic content, nutrients, pathogenic bacteria, and detergents and surfactants. One method that can be used to remove organic and nutrient content is adsorption. The success of the adsorption process is determined by several factors, such as adsorbent type, dosage, contact time, and others. One adsorbent that has attracted the attention of many researchers in recent years is magnetic biochar. This study aims to remove chemical oxygen demand (COD), total suspended solids (TSS), oil, and grease from greywater with a magnetic biochar adsorbent made from palm shells. The study was conducted by varying the dose of 1.2–8.55 g/l and the contact time of 30–150 minutes. The results showed that increasing the dose and contact time can increase the removal efficiency of COD, TSS, oil, and grease. The best results were obtained at a dose of 8.55 g/l and a contact time of 150 minutes. The removal of COD, TSS, oil, and grease was 87.91%, 88.46%, and 99.96%, respectively.

Keywords: adsorption; COD; magnetic biochar; oil and grease; TSS

Introduction

High population growth has an impact on increasing the need for clean water, thereby encouraging people to look for alternative sources of clean water. The source of water from the drilling process requires a large amount of money, and if sourced from rainwater, it cannot be used continuously because of seasonal patterns and ever-changing rainfall. Therefore, one way that can be used is to treat wastewater and reuse it as an alternative source (Skudi et al., 2014).

Greywater is wastewater originating from kitchen sinks, bathrooms, and laundry water (except for wastewater from toilets), which has a pH in the range of 6.1–8.5 (Ghaitidak & Yadav, 2014; Li et al., 2009). According to Shaikh et al., (2015), 55-75% of greywater is produced from total household wastewater. Greywater discharged into the environment without prior treatment causes a decrease in surface water quality because it will be contaminated with organic matter, nutrients, pathogenic bacteria, micro-pollutants, and detergents (Chinyama et al., 2012).

Reusing greywater is one way to prevent negative impacts on the environment, reduce the volume of wastewater, effectively use it to conserve domestic water use, overcome the problem of a shortage of clean water, and reduce the waste water produced by irrigation and flushing toilets (Ghaitidak & Yadav, 2014; Wang et al., 2018). Greywater as toilet flushing water is a very important water sustainability concept, and if utilized, it can reduce water use and pollution arising from offices and industry by 14% and 10%, respectively (Qomariyah, 2016). Several countries have used greywater as water for flushing toilets and watering plants. For example, in Ottawa, Canada, a family uses greywater from a shower, water for washing clothes, and cutlery utensils as water

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for flushing three toilets and watering plants (Handayani, 2013). In general, the characteristics of domestic wastewater are total suspended solids (TSS), chemical oxygen demand (COD), oil and grease, respectively (25–322, 100–700, 22–106 mg/l) (Asadiya & Karnaningroem, 2018; Ghaitidak & Yadav, 2014). This application would also be better implemented in Indonesia to maintain the sustainability of the availability of water resources.

The greywater treatment currently used is the anaerobic-aerobic biofilter. This system has drawbacks such as requiring a residence time of 24 hours, a longer start-up time, and producing sludge (Firmansyah & Razif, 2016). One alternative method that can be used is the adsorption method. Adsorption is the process of absorption of a particular compound molecule by the surface of a solid or liquid substance (Agustina, 2016). Adsorption is a common method used to remove organic and inorganic substances from water (Rashed et al., 2013). The adsorption method using an adsorbent has advantages over other processing methods due to its simple design, low initial production costs, and the fact that it does not require large areas of land. Afrianita et al. (2010) used the adsorption method to remove the COD content contained in domestic wastewater. Rahmawati et al. (2013) set aside the COD and BOD content in laboratory wastewater using the adsorption method. Adsorption is widely used to remove pollutants from waste, especially those that are difficult to degrade (Agustina, 2016). One of the adsorbents that has attracted researchers in recent years is biochar because it has a high adsorption capacity, especially for treating pollutants in water (Deng et al., 2017). Biochar has the capacity to remove contaminants from wastewater, making it ideal for water treatment. The advantage of biochar-based water treatment is that it is a low-cost alternative and produces cleaner treatment (Gwenzi et al., 2017).

Biochar is a solid residue formed from the thermal decomposition of biomass with limited oxygen (Park et al., 2011). The effectiveness and application of biochar depend on the biomass feedstock and the conditions under which it is produced (Zhang et al., 2012). Palm shell is the hardest part of the component found in palm oil that is produced from processing palm kernels and has a calorific value of 3,500–4,100 kcal/kg, which has not been optimally utilized and has the potential to be used as charcoal (Susanto, 2017). The drawback of biochar is its difficulty to separate from water because of its very small size and low density (Zhou et al., 2018).

Magnetic biochar is a modification of biochar where the adsorbent has magnetic properties through the addition of metal ions. The magnetic nature of biochar allows the biochar to be separated from the processing water using a magnet. $FeCl_3.6H_2O$ is a chemical that is generally used to create magnetic properties in biochar because of the non-volatile nature of iron (Fe^{3+}) at high temperatures (Saleh et al., 2016). In addition, the use of $FeCl_3.6H_2O$ can increase the surface area and pore volume of biochar, thereby increasing its capacity (Mubarak et al., 2014). Magnetic biochar produced from waste material shows good adsorption to overcome pollutants in water (Mubarak et al., 2014).

This study aims to synthesize magnetic biochar from palm shells in accordance with SNI 06-3730-1995 and determine the best dose and contact time of magnetic biochar for the removal of COD, TSS, oil and grease in greywater in accordance with Regulation. (Peraturan Menteri Lingkungan Hidup, 2016).

Method

The research flow chart can be seen in Figure 1.

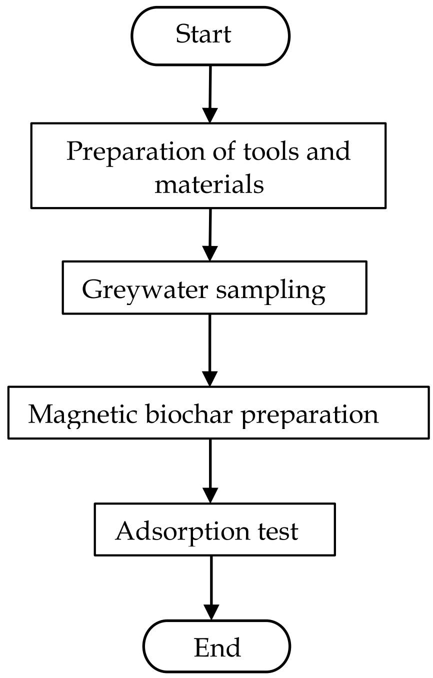

Figure 1. Research flow chart

Wastewater

Greywater was obtained from PT. X with the sampling method referring to SNI 6989 59:2008. Samples were taken at the outlet of the third grease trap. Greywater was tested for parameters of pH, COD, TSS, oil and grease. Sampling points can be seen in Figure 2.

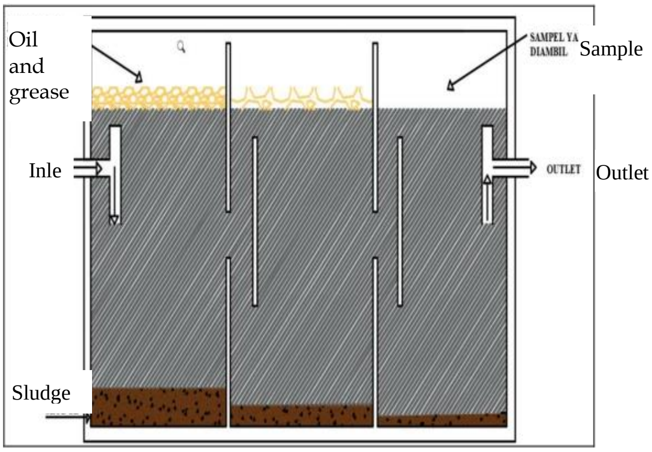

Figure 2. Grease trap and sampling point

Magnetic Biochar Preparation

The palm shells are cleaned and then dried at a temperature of 105°C for 24 hours (Mubarak et al., 2014), and crushed until they pass 100 mesh sieves. The impregnation process uses 150 grams of palm shell. Impregnation was carried out using $FeCl_3.6H_2O$ with an impregnation ratio of 0.5 w/w. Stirring was carried out with a stirring speed of 150 rpm at 30°C for 3 hours. Then it is dried in an oven at a temperature of 100°C for 24 (Mubarak et al., 2014). The dried palm shells were then pyrolyzed at a temperature of 550°C for 20 minutes with a flow of N_2 gas of 0.2 l/minute (Zhang et al., 2014). The magnetic biochar washed with distilled water to neutralize the pH and placed in the oven at 105°C for 3 hours. The resulting magnetic biochar was tested for moisture content, ash content, and carbon content using the American Standard Testing and Material (ASTM) to determine the characteristics of the raw material after it had become magnetic biochar.

Adsorption experiment

Adsorption was initiated by adjusting the pH using 1 M HCl and 1 M NaOH to determine the optimum adsorption pH. The pH used was 5; 6.8; 7. The main research was carried out in a 1000 ml beaker with a working volume of 500 ml. The dose used is 1.25; 3.15; 5; 6.8; and 8.55 g/l (Mubarak et al., 2015), with a contact time of 30; 60; 90; 120; and 150 minutes. The sample was stirred with a jar test at 150 rpm and then allowed to stand for 30 minutes (Ahmad et al., 2009; Azis et al., 2009; and Othman et al., 2013). The supernatant was analyzed with COD, TSS, oil and grease test parameters.

Result and Discussion

Initial Characteristics of Greywater

Sample testing is needed to determine the initial characteristics of greywater. Tests were carried out according to the research parameters as substrates by testing pH, COD, TSS, oil and grease. The results can be seen in Table 1.

Table 1. Greywater Initial Characteristic

Parameter	Value
pH	6.8
COD (mg/L)	397
TSS (mg/L)	260
Oil and grease (mg/L)	152

Based on Table 1, all parameters in greywater are still above the established quality standards. This is because activities that produce greywater, such as kitchen sinks, bathrooms, and laundry water, still contain organic matter, nutrients, micro-pollutants, detergents, or surfactants (Eriksson et al., 2002). High concentrations of COD, TSS, oil and grease from greywater can cause the growth of aquatic biota to slow down due to reduced penetration of sunlight, resulting in a decrease in the process of photosynthesis (Mohammed, 2013). Therefore, greywater requires further processing to reduce the concentration of COD, TSS, oil and grease so that it can be discharged into receiving water bodies and meet the established quality standards.

Characteristics of Magnetic Biochar from Palm Shells

Proximate analysis was carried out to determine the suitability of the quality of magnetic biochar from palm shells made with SNI 06-3730-1995. The results of the magnetic characterization test of biochar from palm shells can be seen in Table 2.

Table 2. Magnetic Biochar Characteristics

Parameter (%)	Value
Water Content	1.13
Ash Content	4.99
Volatile Matter	15.85
Fixed Carbon	78.03

Water Content

The determination of the water content of magnetic biochar from palm shells was carried out at a temperature of 105°C for 1 hour to determine the hygroscopic (moisture) properties of magnetic biochar. The water content can affect the closed pores of magnetic biochar and produce low carbon (Lori et al., 2017). High water content can weaken the adsorption power of activated carbon as an adsorbent. This is influenced by carbon, which is composed of 6 C atoms at each hexagonal angle, allowing water droplets to be trapped inside and partially covering the carbon pores so that the adsorption power of the adsorbent is not maximized (Siregar et al., 2015). According to Bakhtiar et al. (2019), excessive water content will fill the empty space between the biochar and cause the biochar to experience sudden cracks due to the fragility of the structure. Based on Table 2, the moisture content of magnetic biochar from palm shells in this study was 1.13%, which indicated that it met the water content specified in SNI 06-3730-1995, namely a maximum of 15% for powdered activated carbon.

Ash Content

The determination of the value of ash content in magnetic biochar from palm shells was carried out at a temperature of 750°C for 2 hours using a furnace. According to Gumus & Okpeku (2015), ash content is the residue left from the raw material when it undergoes a pyrolysis process. Ash consists of minerals such as silica, aluminum, iron, magnesium, and calcium. The ash contained in activated carbon is considered an impurity because it can block the pores, reducing the surface area and the adsorption capacity (Maulina & Iriansyah, 2018). Based on Table 2, the ash content of magnetic biochar from palm shells in this study was 4.99%, which indicates that magnetic biochar from palm shells met the ash content specified in SNI 06-3730-1995, which is a maximum of 10%. The low ash content indicates that the quality of the raw materials in this study has the potential to be produced into magnetic biochar.

Volatile Matter

Determination of volatile matter value in magnetic biochar from palm kernel shells was conducted at 950°C for 7 minutes using a furnace. Volatile matter is formed from the volatilization process of cellulose and hemicellulose. This happens because the higher the temperature and the smaller the particle size, the more volatile substances are evaporated (Gumus & Okpeku, 2015). The high value of volatile matter indicates that there are still non-carbon compounds attached to the carbon surface, especially nitrogen (N), hydrogen (H), and oxygen (O) atoms that are tightly bound to carbon atoms. These non-carbon compounds are impurities that can cover the surface or biochar pores so that they have an impact on biochar absorption (Pari et al., 2006; Zhang et al., 2014). Based on Table 2, the magnetic volatile matter of biochar from palm shells in this study was 15.85%, which indicates that the magnetic biochar from palm shells has fulfilled the volatile matter specified in SNI 06-3730-1995, which is a maximum of 25%.

Fixed Carbon

The determination of fixed carbon content aims to determine the pure carbon content contained in magnetic biochar. Carbon content (fixed carbon) is the amount of carbon solids in the biomass that is left in the form of char after the pyrolysis process. The higher the carbon content, the fewer impurities are contained in biochar (Zhang et al., 2014), so as to improve the quality of biochar in the adsorption process. The fixed carbon value is obtained from the reduction of the amount of pure carbon contained in biochar minus the values of the water content, ash content, and volatile matter (Basu, 2010). Based on Table 2, the carbon content of magnetic biochar from palm shells in this study was 78.03%, which indicates that magnetic biochar from palm shells has fulfilled the fixed carbon specified in SNI 06-3730-1995, which is at least 65%.

Effect of Dose and Contact Time on Removal Efficiency and Adsorption Capacity of COD

The addition of magnetic biochar doses affects the removal of COD concentrations which can be seen in Figure 3.

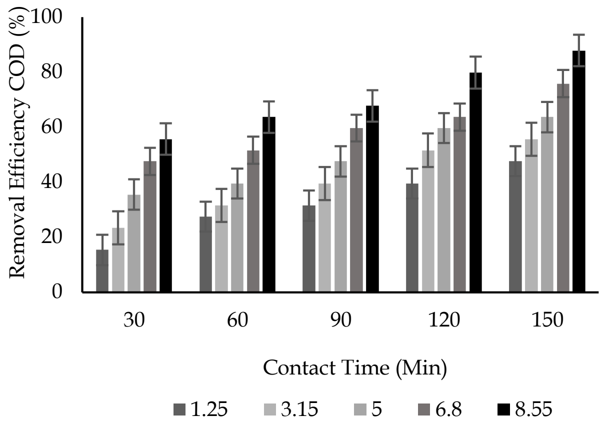

Figure 3. The Effect of Dose and Contact Time of Magnetic Biochar on COD Removal Efficiency

Figure 3 shows the effect of the dose and contact time of magnetic biochar on the removal of COD concentrations. The lowest removal occurred at a dose of 1.25 g/l and a contact time of 30 minutes with a removal efficiency of 15.37%, whereas the highest removal occurred at a dose of 8.55 g/l and a contact time of 150 minutes with an efficiency of 87.91%. The COD concentration decreased with each variation of the dose and contact time of the magnetic biochar used. The same thing also happened in the study of Gaikwad & Mane (2013), where the greater the dose and contact time of magnetic biochar given, the higher the efficiency of removing COD concentrations in greywater. The high efficiency is influenced by the number of doses given so that the available surface area is greater to absorb COD concentrations in the greywater, and the longer contact time causes the process of diffusion and absorption of adsorbate molecules to be more optimal so that the percentage of removal will increase.

The efficiency in this study was higher than that of Gaikwad & Mane (2013) due to the difference in the size of the biomass used, where this study used a 100-mesh size with a small particle size resulting in a large biochar surface area, and the use of $FeCl_3.6H_2O$ could increase the surface area and pore volume in biochar (Mubarak et al., 2014). In carrying out the adsorption process, it will continue as long as the equilibrium point has not been reached. The stagnation point in an adsorption process is the point at which process saturation begins. This saturation can be affected by the number of active sites; the smaller the amount of adsorbate, the greater the desorption process. This saturation process is usually determined based on time, although the time to achieve equilibrium and saturation varies for each adsorption process. This difference is influenced by the type of interaction that occurs between the adsorbent and the adsorbate as well as by the selectivity of the adsorbent on the adsorbate (Wicaksono, 2012). The adsorption results have not reached equilibrium because the active sites available in the adsorbent still allow for the adsorption process to occur due to the large surface area, pore size, and pore volume of the adsorbent (Alimano & Syafila, 2014).

The adsorption process mechanism using magnetic biochar in greywater goes through several stages, namely bulk diffusion on the surface of the particles, which is the transfer of organic matter through the bulk liquid layer to the film layer that coats the adsorbent. The next stage is diffusion through the external carbon surface boundary layer, which is a process of diffusion transfer of organic matter in the stagnant film layer towards the adsorbent pore. These two stages are greatly influenced by the mixing process (Cecen & Aktas, 2012).

The next stage is diffusion in the pore structure, which is the transfer of material to be absorbed through the pores contained in the adsorbent (Bottani & Tascon, 2011; MetCalf & Eddy, 2003). At this stage, pore diffusion occurs when organic matter enters the pores filled with liquid. In addition, surface diffusion occurs when organic matter is absorbed along the surface of the adsorbent. The final stage is the adsorption process, which is the process of attaching the absorbed material to the adsorbent (Cecen & Aktas, 2012).

In addition to removal efficiency, this research also determined the adsorption capacity to determine the amount of COD concentration in greywater that could be adsorbed by each gram of biochar magnetic adsorbent from palm shells. The magnetic adsorption capacity of biochar for COD removal can be seen in Figure 4.

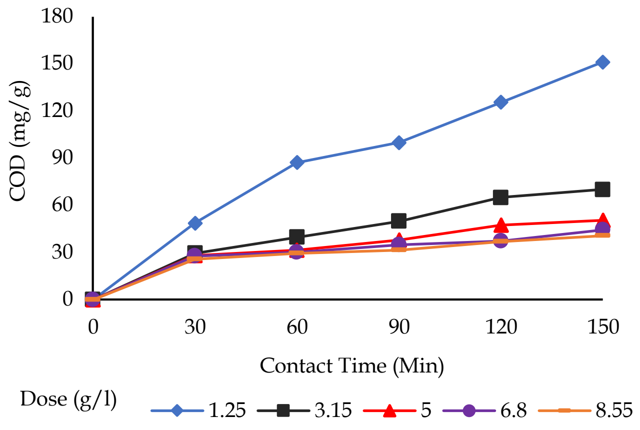

Figure 4. Magnetic Adsorption Capacity of Biochar in COD Removal

The highest magnetic adsorption capacity of biochar was at a dose of 1.25 g/l and a contact time of 150 minutes at 151.20 mg/g. When compared to the study of Afrianita et al. (2010), the adsorption capacity in this study was higher. This is influenced by the contact time, which is a very decisive factor in the adsorption process. According to Cecen & Aktas (2012), a longer contact time allows the processes of diffusion and attachment of adsorbate molecules to take place better. The concentration of organic substances will decrease with increasing contact time, resulting in greater absorption and the ability to adsorb more substances.

In Figure 4, the absorption capacity of each variation can be seen. For a dose of 1.25 g/l and a contact time of 30 minutes, the adsorbent could absorb 48.80 mg/g, while at a dose of 8.55 g/l and a contact time of 30 minutes, the adsorption capacity was 25.85 mg/g. Absorption capacity begins to decrease with an increasing number of doses administered. This decrease was caused by the agglomeration of the adsorbent, so that the surface of the adsorbent was not completely exposed. This causes a reduction in the active surface area of the adsorbent so that the absorption process is ineffective, which results in reduced absorption capacity (Afrianita et al., 2010).

Effect of Dose and Contact Time on Removal Efficiency and Adsorption Capacity of TSS

The addition of magnetic biochar doses affects the removal of TSS concentrations which can be seen in Figure 5.

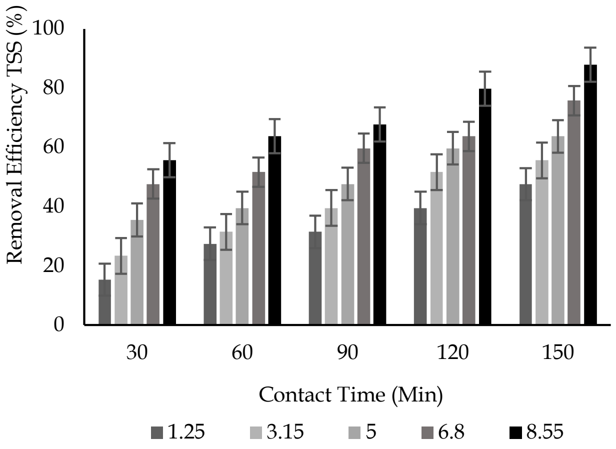

Figure 5. The Effect of Dose and Contact Time of Magnetic Biochar on TSS Removal Efficiency

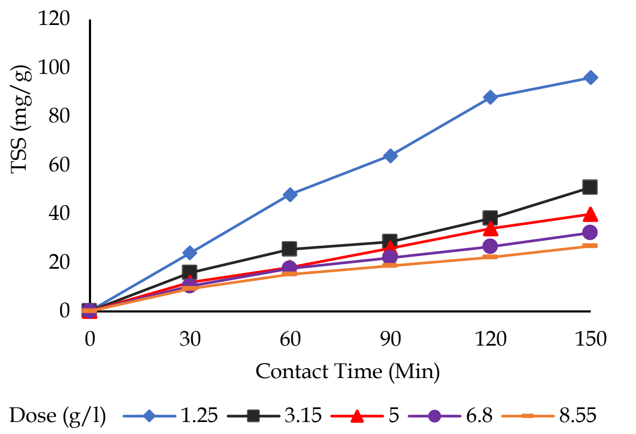

Figure 6. Magnetic Adsorption Capacity of Biochar in TSS Removal

Based on Figure 5, the lowest removal of TSS occurred at a dose of 1.25 g/l and a contact time of 30 minutes with a removal efficiency of 11.54%, while the highest removal was at a dose of 8.55 g/l and a contact time of 150 minutes with a removal efficiency of 88.4%. The concentration of TSS decreased with each variation of the dose and contact time of the magnetic biochar used. This is due to an increase in the adsorbent dose resulting in an increase in the number of active pore sites available for adsorption. Adsorbent dosage is a factor that generally influences adsorption success (Said et al., 2015) because it plays a role in determining the adsorbent capacity and adsorption mechanism that depend on the surface of the available adsorbent. This increase will continue for some time until the increase becomes almost constant because the active site has been occupied and there is no further adsorption (Anijiofor et al., 2018).

In this study, the best TSS allowance was 88.46%. The percentage of removal results is lower compared to the study of Said et al. (2015). This is due to the slower stirring speed of 150 rpm which causes only a slight reduction in the resistance of the film layer, making it difficult for the particles to stick to and be adsorbed on the surface of the adsorbent. High stirring speeds can reduce the resistance of the film surface and cause the particles to change into smaller sizes so that they will easily stick and be adsorbed on the surface of the adsorbent (Said et al., 2015).

In addition to removal efficiency, this research also determined the adsorption capacity to determine the amount of TSS in greywater that could be adsorbed by each gram of biochar magnetic adsorbent from palm shells. The magnetic adsorption capacity of biochar for TSS can be seen in Figure 6.

Based on Figure 6, the highest biochar magnetic adsorption capacity was found at a dose of 1.25 g/l and a contact time of 150 minutes at 96 mg/g. The adsorption capacity in this study was higher than the research of Shahawy & Heikal (2018), where the contact time used in this study was longer. Contact time is very decisive in the adsorption process because it determines the time needed for the adsorbate to reach equilibrium (Shahawy & Heikal 2018).

The adsorption capacity decreases with increasing doses of adsorbent used but increases with the length of contact time used. For a dose of 1.25 g/l and a contact time of 30 minutes, the adsorbent could absorb 24 mg/g, while at a dose of 8.55 g/l and a contact time of 30 minutes, the adsorption capacity was 9.36 mg/g. The adsorption capacity indicates the amount of adsorbate adsorbed per unit dose of adsorbent, so its value is affected by the amount of adsorbent dose. If the adsorbent dose increases while the adsorption contact time and adsorbate concentration remain constant, the increase in surface area will increase the spread of the adsorbate, so that per unit dose of adsorbent, it does not fully adsorb the adsorbate (Kurniawan, 2011).

Effect of Dose and Contact Time on Removal Efficiency and Adsorption Capacity of Oil and Grease

The addition of magnetic biochar doses affects the removal of oil and grease concentrations in greywater, as shown in Figure 7.

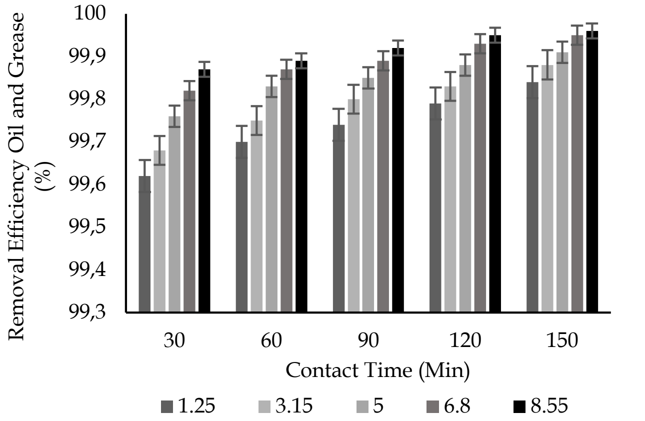

Figure 7. The Effect of Dose and Contact Time of Magnetic Biochar on Oil and Grease Removal Efficiency

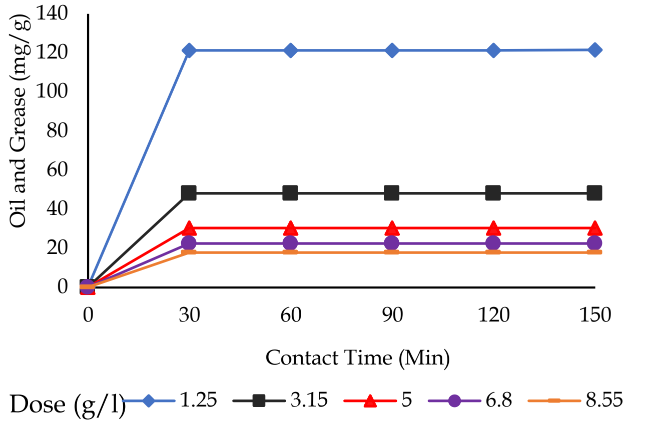

Figure 8. Magnetic Adsorption Capacity of Biochar in Oil and Grease Removal

Based on Figure 7, the lowest oil and grease removal occurred at a dose of 1.25 g/l and a contact time of 30 minutes with a removal efficiency of 99.62%, while the highest removal occurred at a dose of 8.55 g/l and a contact time of 150 minutes with a removal efficiency of 99.96%. The best results from this study can remove oil and grease by 99.96%. The decrease in oil and grease concentrations occurs because free greasety acid particles are adsorbed by the pores on the carbon surface (Shahawy & Heikal, 2018). Activated carbon is hydrophobic, so it easily absorbs substances or solutions such as oil containing organic compounds such as free greasety acids. In the adsorption process between magnetic biochar and free greasety acids, which are negatively charged, it causes an attractive force with positively charged active sites on the surface of the adsorbent (Shahawy & Heikal, 2018).

The removal rate in this study was higher than that in the study by Abuzar et al. (2012) due to differences in the manufacturing process and raw materials used. In this study, the adsorbent used underwent a pyrolysis process, so it has a larger surface area and high porosity. The larger surface area and high porosity cause the adsorbent to work optimally along with the contact time because the adsorbent and adsorbate are allowed to interact with each other so that the opportunity for the adsorbate to fill on each surface and the magnetic properties added to the biochar increase the surface area and pore volume on the biochar, so that the adsorption process increases and provides effectiveness during processing (Mubarak et al., 2014; Zainol et al., 2014).

This research also determined the adsorption capacity to determine the amount of oil and grease in greywater that could be adsorbed by each gram of biochar magnetic adsorbent from palm shells, as shown in Figure 8.

Based on Figure 8, the highest magnetic adsorption capacity of biochar was at a dose of 1.25 g/l and a contact time of 150 minutes at 121.41 mg/g. The adsorption capacity in this study was higher than that of Abuzar et al. (2012) due to the longer contact time used. Contact time has an influence on the absorption capacity of oil and grease, and longer contact time provides a greater opportunity for the adsorbate to be adsorbed by the adsorbent. At the initial time where the adsorbate has not been adsorbed, it will be possible to be adsorbed at a later time when the adsorbate will move by rotating turbulence to look for empty space. In terms of fulfilling the pore volume, the longer the contact time, the more it will allow the adsorbate to be pushed to the end of the pore and provide pore space to be filled by new adsorbate (Alimano & Syafila, 2014).

In Figure 8, the absorption capacity of each variation can be seen. For a dose of 1.25 g/l and a contact time of 30 minutes, the adsorbent could absorb 121.13 mg/g, while at a dose of 8.55 g/l and a contact time of 30 minutes, the adsorption capacity was 17.75 mg/g. Absorption capacity begins to decrease with an increasing number of doses administered. The adsorption capacity shows the amount of adsorbate adsorbed per unit weight of the adsorbent. Therefore, the value of the adsorption capacity is affected by the dose of the adsorbent. If the dose of the adsorbent is getting bigger while the contact time and concentration of the adsorbate are fixed, it will cause the spread of the adsorbate so that per unit gram of adsorbent, it does not fully adsorb the adsorbate (Kurniawan, 2011). This causes the adsorbent to not work optimally.

Conclusion

Magnetic biochar has been successfully synthesized from oil palm shells. The results showed that magnetic biochar synthesized from oil palm shells can remove

COD, oil and grease, and TSS from greywater with an efficiency of 87.91%, 99.96%, and 88.46% respectively. The best results were obtained at a dose of 8.55 g/l and a contact time of 150 minutes.

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Author Contributions
Authors listed in this article contributed to the research and development of the article. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest
The authors declare no conflict of interest.

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