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Original Article

Evaluation of Some Eco-Friendly Food Residues for Removal of Methylene Blue Dye from Aqueous Solutions

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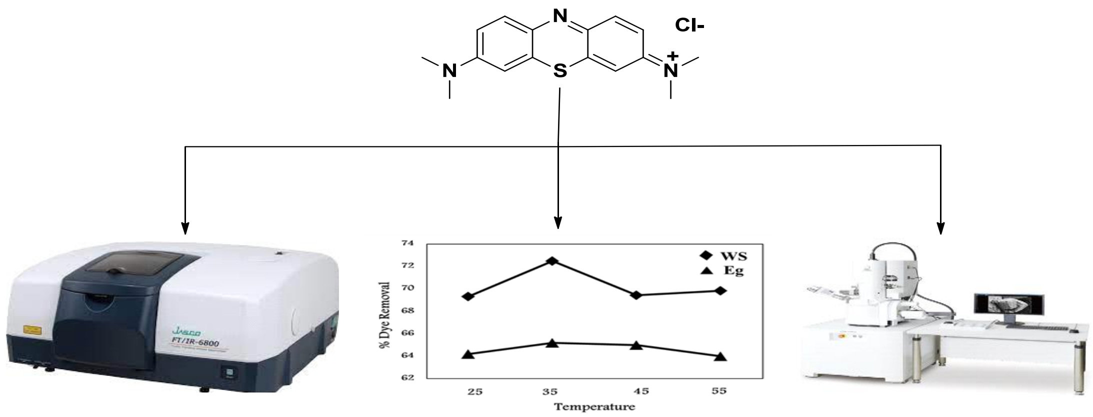
ABSTRACT

Walnut shell (WS) powder and eggshell (Eg) powder were grounded and sieved using a 75 µm hole diameter sieve. Both WS and Eg powders were characterized by SEM and IR technologies, and their adsorption capacity was tested towards Methylene blue dye (MB). The tested parameters for MB removal efficiency (RE) were contact time, initial dye concentration, and temperature. It was found that RE of 20 mg/L MB equaled 65.86% after 10 min and 64.82 % after 30 min in the presence of WS and Eg, respectively. For 20, 40, 60, 80 and 100 mg/L of MB, RE using WS equalled to 65.87%, 71%, 71.8%, 72% and 72.34 respectively. However, using Eg, RE for the same concentrations of MB was noticed to be 60.01%, 55.23%, 51.62%, 46.01%, and 44.52%, respectively. At varing temperatures, for 20 mg/L of MB, the RE was 69.34%, 72.5%, 69.44%, and 69.85% at 25, 35, 45, and 55°C on order using WS powder. While at the same temperatures, RE was 64.21%, 65.21%, 65%, and 64%, respectively, using Eg powder. It can be concluded that WS powder has shown greater adsorption capacities towards MB than Eg powder.

GRAPHICALABSTRACT



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Introduction

Discharging synthetic dyes into the environment has created a big challenge for scientists interested in environmental treatment field [1]. The number of synthetic dyes consumed in commercially important industries has been reaching higher rates recently. This is because manufacturing materials like paper production, cosmetic, textiles industry, pharmaceutical, and painting technologies are referred to as extensively consumed industries for these dyes [2]. In addition, these kinds of industries keep growing every year, together with dyes consumed by the increasing global population. Disposal of industrial wastewater near water sources is affecting the environment and causing a huge water pollution problem as dyes are toxic and hazardous and influence penetrated sunlight that is required for all life aspects [3]. Methylene blue (MB) is a commonly used dye in different industries [4]. Its role is to impart color to cotton, wool, paper, and silk products. The color of MB in water is dark blue, and the structure is given as an aniline-based basic dye ($C_{16}H_{18}ClN_3S$) Figure 1 [4]. The limit of MB used in such industries exceeds the recommended value. Exceeding the limit may cause a serious health problem when discharged to water sources nearby. According to previous studies, MB might cause short or hard breathing when inhaled by accident. If taken by mouth, it might cause a burning sensation, vomiting, sweating, mental confusion, and nausea. It has also been stated that exposure to a large amount of MB could result in bad headaches and chest and abdominal pain. The above risks and consequences of using MB in the industry sector created an urgent need to remove it from water bodies and protect the well-being of different life aspects.

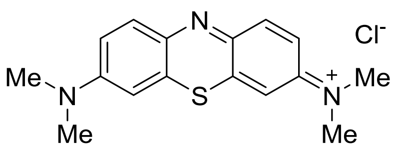

Figure 1: Chemical structure of Methylene blue (MB) dye [5]

In order to treat polluted aqua systems, traditional methods were introduced and carried out for dyes removal, including biological, physical, and chemical methods, including but not limited to activated carbon, photocatalytic degradation, filtration, ozonation, and biodegradation. However, these technologies were costly, complicated, and had other environmental concerns [6]. The adsorption method has been introduced as a promising technology to solve wastewater problems efficiently and effectively and also overcame drawbacks indicated by previous methods [7]. Adsorption technology uses simple and inexpensive substances suitable for the adsorption of organic and inorganic toxic wastes and compatibility with nature. A number of adsorbents have been performed for wastewater treatment, such as biomass and agricultural by-products [7]. The walnut is one of the most commonly spread trees and an economically important plant. About three million tons of walnut has been produced over the universal stage since 2012 [8]. In addition to its high nutrition benefits, by-products obtained from walnut fruit, like shell and husk, are greatly valuable compounds containing phenolics [9]. Many studies reveal that using walnut shell WS is an excellent alternative and attractive material for removing carcinogenic compounds by adsorption due to affordability and high efficiency [10-12]. It can also be used as an antioxidant and antimicrobial compound, which has gained considerable attention in medical applications, especially, for treating inflamed skin, ulcers, and hyperhidrosis [13, 14]. Producing adsorbents through the transformation of waste material has helped obtain the useful product for industrial and scientific research [4]. The excellent adsorptive capability was seen by a walnut shell towards a number of organic compounds [15], dyes [16], thallium [17], and heavy metals [15]. Eggshell Eg waste is another example of adsorbents that were used in multiple applications such as additives and fertilizers as well as adsorbents for heavy metals. Calcium carbonate represents the major chemical component of eggshells in addition to calcium phosphate and magnesium carbonate [18, 19]. The eggshell structure is porous, making it an excellent selection to serve as an adsorbent. Moreover, the eggshell powder is affordable, as noted by restaurants and food-producing industries' high daily consumption of eggs [20]. This paper aims to compare two cost-effective adsorbents for the treatment of water contaminated with low levels of MB. Walnut shell powder and eggshell powder were chosen for this purpose. The powder of each plant was characterized by Fourier-transform infrared spectroscopy (FT-IR) and Scanning Electron Microscopy technique (SEM). Different parameters were examined, including initial dye concentration, temperature, and contact time. The examination of particles was done using UV-visible technology.

Materials and Methods

Walnut plants and eggs (adsorbents) were purchased from a local store. 1000 mg/L of Methylene blue MB ($C_{16}H_{18}ClN_3S$) (molecular weight= 319.85 g/mol) (adsorbate) was used as received. Different glass wear was used during lab work, measuring cylinders, conical flasks, thermometers, blenders, and electronic weighing.

Preparation of walnut shell and egg shell micro-powders

First, the original walnut plant and egg shells were cracked separately into small parts and washed several times with a water tap. Then, they were washed with distilled water to eliminate impurities and dusty particles. Later on, the cracked walnut and eggshell were heated up in the oven at 105 °C to be entirely dry. The plant was then ground at home using a home grinder. The ground powders were sieved individually using a 75 μm sieve (hole's diameter). Both powders are ready now to be used for dye adsorption experiments.

Preparation of MB stock solutions

All solutions were prepared using double distilled water. A stock solution was firstly prepared by adding 1 g of MB powder into 1L double distilled water (1000 mg/ 1L). The dye was stirred until it was completely dissolved. The experimental solution was prepared by diluting a definite volume of the stock solution to get the desired concentration. Five standards of dye solution with different dye concentrations (20, 40, 60, 80, and 100 mg/L) were prepared.

Kinetic and removal experiments

To measure removal efficiencies, 0.1 g of WS and Eg adsorbent was added to MB solutions (20 mg, 40 mg, 60 mg, 80 mg, and 100 mg) and then exposed to shaking using a shaking water bath. The blank and standard solutions were filtrated, and the concentration of MB dye removal percentage was calculated by the UV-visible spectrophotometer at a wavelength of 565 nm. The obtained absorbance of the blank and the standards were recorded. Table 1 shows the characteristic of Methylene blue dye.

Table 1: Characteristics of methylene blue dye

Dye Chemical formula	Molecular weight (g/ mol)	Mp °C	Type of dye	bp	λ_{max} (nm)
Methylene blue $C_{14}H_{18}N_3Scl$	319.85	100-110	Basic blue pH (9.4-14.0)	decomposition	665

Effect of contact time

0.1 g of walnut shell and eggshell powder were added separately to four tubes containing 10 mL of MB with a concentration of 20 mg/L at room temperature. Firstly, the mixture inside each tube was stirred continuously with a magnetic bar for specifically determined times. The samples were then filtrated, as explained in Table 2.

After filtration, liquid parts were collected and tested with a UV-VIS spectrophotometer to investigate color removal efficiency at specific contact times.

Effect of initial concentration

The initial concentration of MB (the adsorbent) solution has a deep influence and is very important when studying dye removal. Different initial concentrations of MB dye were chosen (20, 40, 60, 80, and 100 mg/L) to investigate the effect of changing dye concentration on dye removal using walnut shell powder or eggshell powder separately, as listed in Table 3.

Table 2: Effect of contact time on dye removal efficiency using different adsorbents (A) walnut shell powder (B) eggshell powder

(A) Adsorbent	Particles size	Con. of adsorbate	Tube No.	V. of adsorbate	Reaction time till filtration
WS	75 µm	20 mg/L	Tube 1	10 mL	10 minutes
			Tube 2	10 mL	30 minutes
			Tube 3	10 mL	60 minutes
			Tube 4	10 mL	90 minutes
(B) Adsorbent	Particles size	Con. of adsorbate	Tube No.	V. of adsorbate	Reaction time till filtration
Eg	75 µm	20 mg/L	Tube No.	V. of adsorbate	Reaction time till filtration
			Tube 1	10 mL	10 minutes
			Tube 2	10 mL	30 minutes
			Tube 3	10 mL	60 minutes
			Tube 4	10 mL	90 minutes

Table 3: Effect of initial dye concentration on dye removal using different adsorbents (A) walnut shell powder (B) eggshell powder

(A) Adsorbent	Particles size	Con. of adsorbate	Tube No.	V. of adsorbate
WS	75 µm	20 mg/L	Tube 1	10 mL
		40 mg/L	Tube 2	10 mL
		60 mg/L	Tube 3	10 mL
		80 mg/L	Tube 4	10 mL
		100 mg/L	Tube 5	10 mL
(B) Adsorbent	Particles size	Con. of adsorbate	Tube No.	V. of adsorbate
Eg	75 µm	20 mg/L	Tube No.	V. of adsorbate
		40 mg/L	Tube 1	10 mL
		60 mg/L	Tube 2	10 mL
		80 mg/L	Tube 3	10 mL
		100 mg/L	Tube 4	10 mL

Effect of temperature

Dye removal was also carried out at set temperatures. They ranged from room temperature up to 55 °C. This part aims to study the adsorbent's performance in different temperatures. Eight samples of MB at 20 mg/L were prepared using eight separate tubes. Each tube contained 10 mL of MB selected solution plus 0.1 g of walnut shell powder for the first four tubes and 0.1 g of eggshell powder for the other four tubes. It is worth mentioning that all prepared mixtures were stirred using a magnetic bar during reaction time. Tested temperatures were 25, 35, 45, and 55 °C, as seen in Table 4. The mixtures were then filtered, and the obtained liquid parts were tested with a UV-VIS spectrophotometer to investigate color removal efficiency at specific temperatures.

Characterization

The chemical structure of pristine walnut shell and eggshell powders was analysed using Fourier Transform Infrared (FT-IR) Spectrometer (Shimadzu, Japan). Surface morphology was also studied using Scanning Electron Microscope (SEM) (TESCAN MIRA3 FRENCH).

Table 4: Effect of temperature on dye removal using different adsorbents (A) walnut shell powder (B) egg shell powder

(A) Adsorbent	Particles size	Con. of adsorbate	Tube No.	V. of adsorbate	Reaction temperature
WS	75 µm	20 mg/L	Tube 1	10 mL	25 °C
			Tube 2	10 mL	35 °C
			Tube 3	10 mL	45 °C
			Tube 4	10 mL	55 °C
(B) Adsorbent	Particles size	Con. of adsorbate	Tube No.	V. of adsorbate	Reaction temperature
Eg	75 µm	20 mg/L	Tube 1	10 mL	25 °C
			Tube 2	10 mL	35 °C
			Tube 3	10 mL	45 °C
			Tube 4	10 mL	55 °C

Results and Discussion

The SEM images are shown in Figure 2a and b to observe the morphology of the walnut shell surface. Due to SEM analysis, the surface texture of raw walnut shell is mainly represented by surface folds and pore existence [21, 22]. As clearly seen in SEM images, the rode–like appearance can be attributed to a cellulosic structure where lignin, cellulose, and hemicellulose are the main components [21, 22]]. The rough surface is seen with a number of random cavities. This is due to the pores nature of the walnut shell surface [15, 21, 22]]. These features demonstrate the high porosity of walnut shell surface, which confirms its surface's capability to adsorb MB in its cavities.

The main peaks noticed at the FT-IR spectrum of the raw walnut shell are 3434, 2925, and 1643 cm^{-1}. For stretching vibration of O-H, a broad peak is seen at 3434 cm^{-1}. For C-H stretching vibration found as an alkane, a clear peak is seen at 2925 cm^{-1}. For C=O, stretching belongs to the carbonyl group is seen at 1743 cm^{-1}. Moreover, 1634 cm^{-1} stretching belongs to alkene (Figure 3) [4, 15, 21].

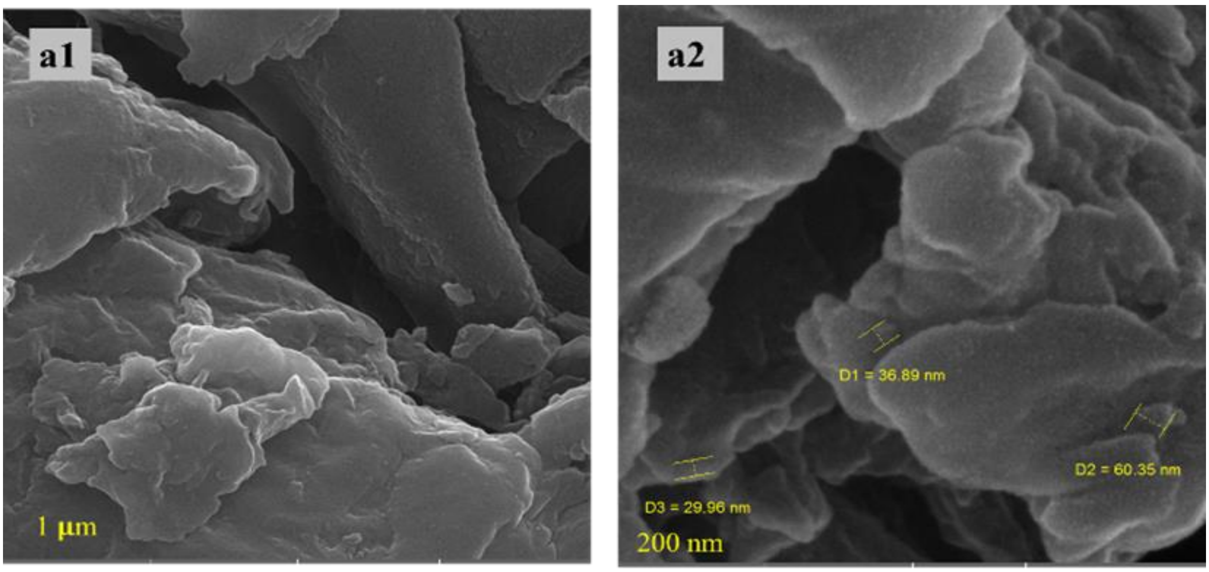

Figure 2: SEM images of raw walnut shell powder

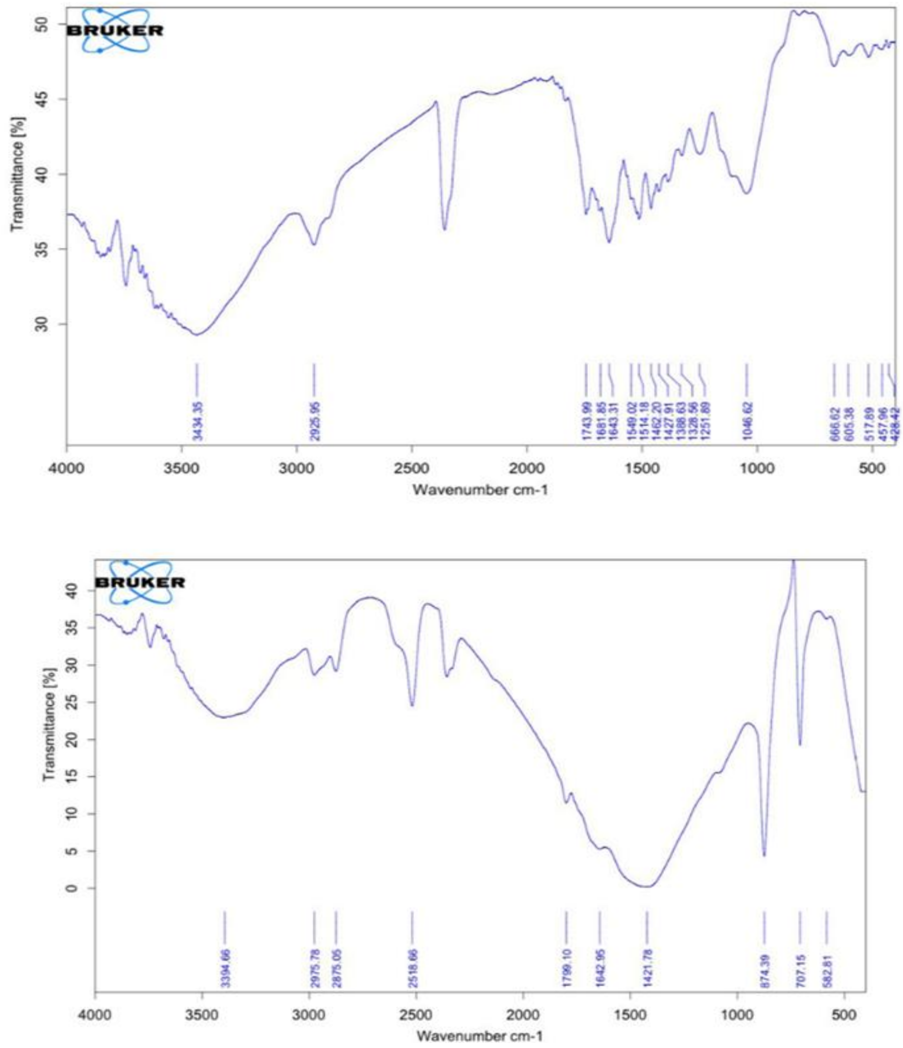

Figure 3: FT-IR spectrum of (a) raw walnut shell powder, (b) raw egg shell powder

Figure 3 shows the FT-IR spectrum, which was recorded for raw egg shell in the range of 500–4000 cm^{-1}. Sharp peaks were noticed at 707, 874, 1421, and 2518 cm^{-1}; a broad peak was also noticed at 3394 cm^{-1} , which is attributed to the absorption of $CaCO_3$ [24]. Peaks at 1799, 2875, and 2925 cm^{-1} attribute to C=O. The broad and strong peak at 1421 cm^{-1} is attributed to C=O bonds. Moreover, the peak at 582 cm^{-1} belongs to Ca–O bonds [25, 26].

Adsorption studies (batch experiments)

All the adsorption studies of MB ions carried out by (WS and Eg) powders with the higher surface area are suggested in the current study for better adsorption behaviour. Batch experiments are applied herein to evaluate the adsorption performance of WS and Eg powders. The removal efficiency (R%) and adsorption capacity (q_e (mg/g)) were calculated as follows [27]:

$$R\% = (C_0 - C_e) / C_0 \times 100$$

$$q_e = (C_0 - C_e)\, V / m$$

C_o and C_e are initial and equilibrium concentrations (mg/L) of dye, V is the volume of solution (L), m the adsorbent's weight (g).

Effect of contact time

Four samples were taken from 20 mg MB concentration to study the effect of varied contact time on dye removal percentage in the presence of an adsorbent. After shaking samples at room temperature using a shaking water bath, WS and Eg were added separately to the previously mentioned concentrations. Samples were then filtered and waited until the liquid part gathered a good amount for the next step. Later, the liquid obtained from the filtration was analysed by UV-Vis spectrophotometer at wavelength= 565 nm. The data were a plot to study the removal percentage of the dye. It can be seen that the removal percentage reached nearly 65% in the case of WS powder adsorbent, as presented in Figure 4. Herein, Table 5 shows removal efficiencies of MB at different times using both adsorbates.

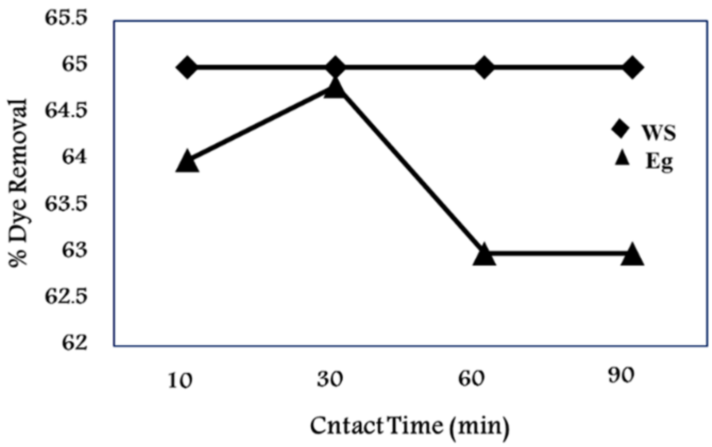

Figure 4: Removal efficiencies of MB at varied contact times using WS powder and Eg powder as adsorbents separately

Table 5: Removal efficiencies of MB at varied contact times using WS and Eg

Con. (mg/L)	RE% of MB by WS	RE% of MB by Eg	Contact Time (min)
20	65.86	64.01	10
	65.12	64.82	30
	65.44	63.30	60
	65.52	63.08	90

The dye removal percentage stayed constant as the time extended from 10 min to 90 min. This can be explained, the free sites at the surface of WS have reached saturation status by MB ions after 10 min. On the other hand, when Eg was used as an adsorbent, the dye removal percentage increased from 10 min to 30 min, as can be seen in Figure 4. It was found that 64.8 % was recorded as the highest dye removal percentage by Eg powder. This value became slightly lower as the time extended from 30 min to 90 min and stayed constant also.

Effect of initial dye concentration

Five samples of varying concentrations ranging from 20 mg to 100 mg per liter by adding 1 g of MB dye to 1 L of the prepared solution were tested, as can be seen in Figure 5. From the results, MB dye was removed rapidly in the first 10 min and started to maintain a constant value around 30 min. This trend can be seen clearly in Figure 5. The dye removal percentage increased when the MB concentration increased too. The trend on increasing the dye removal was as follow 65.87%, 71.00%, 71.81%, 72.00% and 72.34% respectively [28]. However, the dye removal efficiency fluctuated and showed a downward trend ranging from 60.01% to 44.52% using Eg powder as an adsorbent. Table 6 reveals the removal efficiencies of different concentrations of MB using both adsorbates.

Table 6: Removal efficiencies of varied concentrations of MB using WS and Eg

Con. (mg/L)	RE% of MB by WS	RE% of MB by Eg	Contact Time (min)
20	65.87	60.01	10
40	71.00	55.23	
60	71.81	51.62	
80	72.00	46.01	
100	72.34	44.52	

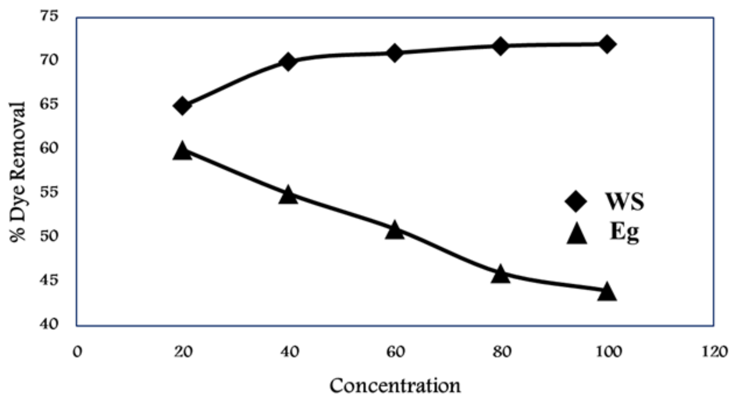

Figure 5: Removal efficiencies of MB at varied concentrations using WS powder and Eg powder as adsorbents separately

Effect of temperature

Figure 6 illustrates the effect of temperature on dye removal efficiency. The tested temperatures were: 25 °C, 35 °C, 45 °C and 55 °C at 20 mg/L dye concentration. The removal efficiency was as followed: 69.34 %, 72.5 5, 69.44 % and 69.85 % respectively. The dye adsorption increased between 25-35 °C and a steady drop between 45-55 °C. Increasing the adsorption at 35 °C enhances the affinity between WS powder and MB dye molecules. This may happen due to the powder's suitability of providing good driving force towards MB molecules. In return, more dye molecules adsorbed numbers moved towards WS powder to interact with its structure. In fact, elevating the temperature may increase the penetration of MB molecules to the WS powder surface, and which is why the adsorption increased at first. After raising temperature more to 45 °C or even 55 °C, the number of removed MB molecules declined. This phenomenon could be due to possible damage to the WS structure at higher degrees, resulting in the failure of WS capability to act as an adsorbent. Table 7 introduces the removal efficiencies of MB at different temperatures using both adsorbates.

Table 7: Removal efficiencies of MB at varying temperatures using WS and Eg

Con. (mg/L)	RE% of MB (by WS)	RE% of MB (by Eg)	Tem. (C)
20	69.34	64.21	25
	72.5	65.21	35
	69.44	65	45
	69.85	64	55

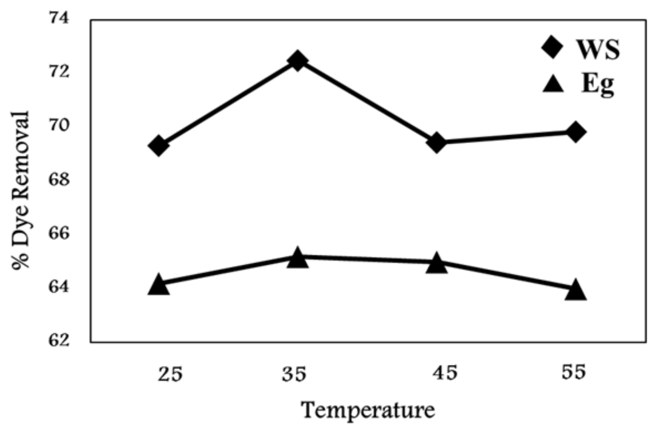

Figure 6: Removal efficiencies of MB at varying temperatures using WS and Eg powder as adsorbents separately

Conclusion

Walnut shell and eggshell powder were perfectly washed and finely ground, then sieved using a 75 μm hole diameter sieve. The collected powders were used to remove methylene blue ions from water and proved to be effective. The tested parameters were as follow: varied contact time, varied initial concentration of dye, and varying temperatures. The removal efficiency of MB using walnut shell powder recorded higher percentages than eggshell powder. After 10 mins, the removal efficiency of MB was 65.86 % using walnut shell powder. However, eggshell powder acquired 30 min to remove 64.82 % of MB. Moreover, removal efficiency at varying concentrations of MB was increasing steadily using walnut shell powder. However, a clear decrease was noticed in the removal efficiency of MB using eggshell powder. At varying temperatures, the removal efficiency was elevated between 25 °C to 35 °C, then declined. This behavior was clearly shown using both powders separately.

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Authors' contributions

All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

Conflict of Interest

The author declared that they have no conflict of interest.

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