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RESEARCH ARTICLE

Acid-activated carbon from Coconut Shell waste for effective removal of Toxic Malachite green dye from aqueous medium and study of regenerative Cycles.

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ABSTRACT

A preparation of activated carbon from coconut shell residue, activated by phosphoric acid. By controlling the ratio of phosphoric acid to coconut shell mass and activation temperature, the textural property and surface chemistry of the prepared activated carbon could be optimized. Characterization results of FESEM and TEM showed that the as-prepared AC had a perfect porous structure and rich surface functional groups. The moderate acid dosages were conducive to the creation of a well-developed porous structure and the incorporation of favorable acidic surface function groups, which is beneficial for excellent adsorption capability. The optimal phosphoric acid loading also reduced the effectiveness of activation if the loading was too high. The over-acidification of the biomass led to extensive crosslinking and the collapse or blockage of micropores, resulting in a decrease in specific surface area and pore volume. This also led to a reduction in the adsorption of the activated carbon. The activated carbon achieved a high adsorption capacity for malachite green (MG), reaching 90.88%. The reuse of the adsorbent was studied by regeneration with three types of eluents: deionised water (neutral), 0.1 M NaOH (alkaline), and 0.1 M HCl (acidic). Among the three, the acidic regeneration agent (HCl) was the most effective in restoring the adsorption performance, surface features, and structural integrity of active sites during the recycling operation. These results highlight the potential of phosphoric acid-activated coconut shell as a sustainable and efficient material for large-scale treatment of dye wastewater, as well as the valorisation of agricultural residues.

Keywords: Adsorption; dye; malachite green (MG); mechanism, agricultural waste; regeneration

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1. Introduction

Synthetic dyes are a common type of chemical compound primarily used in industries such as textiles, paper, plastics, leather, cosmetics, and food processing. These dyes are commonly classified according to their chemical structure and method of application, and are divided into major categories, including azo, anthraquinone, triphenylmethane, and reactive dyes. Each class possesses distinct chromophoric systems that render them colorful and stable, and therefore commercially attractive but environmentally persistent[1-3].

Malachite green (MG); a cationic triphenylmethane dye, is one of the widely used dyes which has been massively adopted into the textile industry, aquaculture and as a biological stain in laboratories. It is very effective and inexpensive, but it is also a known

environmental and health hazard. Because ACP is toxic and mutagenic, it can lead to the accumulation of ACP in aquatic organisms, with serious ecological and human health risks, even at a low concentration. Owing to high solubility and biodegradation resistance, dyes such as MG are refractory in industrial effluents for conventional wastewater treatment processes. Several advanced techniques have been investigated to overcome this problem, including biological breakdown, coagulation-flocculation, membrane filtration and advanced oxidation processes[4-7]. Nevertheless, the adsorption process with AC is one of the most efficient, economical, and commonly used processes, as it is highly effective and provides simplicity, removing a wide range of dyes without producing any harmful by-products. In the current work, efficient removal of Congo red from aqueous solutions using an eco-friendly activated carbon prepared from discarded natural Pterocarpus erinaceus sawdust was described. Adsorbent activation conditions and regeneration techniques should be deemed a significant factor for greener wastewater treatment[8-10].

Agricultural waste, produced in excessive amounts worldwide, is a readily available, low-cost, and renewable resource for environmental applications. These agro-residues, which include coconut shells, corncobs, rice husks, sugarcane bagasse, and various fruit peels, are known to be abundant in lignocellulosic composition and potential precursors for activated carbon production. Conversion of such biomass into active adsorbent materials is not just a way of environmentally friendly waste management, but also in line with the concept of circular economy, that is, the conversion of worthless waste into a valuable commodity[11, 12]. Agricultural waste-derived activated carbon has received increasing interest owing to its high surface area, abundant porosity, and modifiable surface chemistry. These properties make it a useful material for scavenging a variety of pollutants, including heavy metals, dyes, pesticides, and organic contaminants, from both water and air. Activation of the carbonised substances generally comprises physical or chemical procedures that enhance the adsorption properties of the activated carbon[13, 14]. The utilisation of agricultural waste for the production of activated carbon not only can help to alleviate disposal problems caused by agro-waste, but also allows researchers to develop GMO on a cheaper and sustainable process for various treatment technologies. This method is ideal for long-term pollution monitoring, particularly in the treatment of industrial wastewater and contaminated effluents[2, 15].

This investigation highlights the utilisation of agricultural waste such as coconut shell biomass for the preparation of high-performance activated carbon using phosphoric acid-aided thermal activation. The three-dimensional, porous, and high surface area of the optimal material were responsible for the relatively high adsorption capacity of the hazardous dyes (e.g., malachite green). The findings also emphasised the importance of activation conditions, in particular acid dose and temperature, for structural and functional performances of the finished adsorbent. Regeneration tests demonstrated the maintained extensive reusability of the activated carbon, and acidic treatment was found to be the most effective reactivating method for the recovery of adsorption capacity. These results not only highlight the applicability of the agro-waste-based AC as a low-cost and environmentally friendly adsorbent but also provide practical wisdom for sustainable wastewater treatment technology. In general, valorising agricultural residues has the potential to have an environmental impact and recover resources.

2. Experimental part

2.1. Production of activated carbon from coconut shells

Raw coconuts were collected from a local market in Hilla City, Iraq. Fruits were handpicked, and only the hard endocarp shell was collected for processing. The shells were rinsed thoroughly with tap water to remove attached dirt and surface impurities, and then with distilled water. The washed shells were oven-dried at 105 °C to constant weight. Then the dried coconut shells were impregnated with 40 mass% of H_3PO_4 at several mass ratios of acid to shell residue, (i) 1.1, (ii) 1.5:1, (iii) 2:1, (iv) 2.5:1 and (v) 3.0. The soaking time was 12 h for sufficient chemical penetration. The impregnated samples were dried in an oven at 80°C

for 8h. Furthermore, the blank-disc samples were heated to 500°C using a tubular furnace under a N_2 flow for 2 hours, with a heating rate of 10°C/min, for chemical activation. The activated materials were washed with deionised water several times until the pH became neutral, thereby eliminating the remaining chemicals after activation. Methanol was then evaporated under reduced pressure, and samples were dried at 120°C for 6 h. The as-prepared activated carbon was named AC-H_3PO_4.As shown in **Figure 1**.

Figure 1. Production of activated carbon from coconut shells

2.2. Adsorption study

Adsorption experiments were conducted using 100 mL of Malachite Green (MG) dye solution at an initial concentration of 100 mg/L. The solutions were transferred into 100 mL Erlenmeyer flasks, and a 0.01-0.1 g mass of activated carbon was added to each flask. The mixtures were then agitated in a mechanical shaker at room temperature for 1 hour to ensure equilibrium was reached. The initial pH of the dye solutions was approximately 7 and was maintained throughout the experiment. Additional experiments were conducted to investigate the effects of pH (2-10) and temperature (15-36°C) on the adsorption process. Before analysis, all samples were centrifuged to eliminate interference from fine carbon particles. The residual dye concentration in each sample was determined using a UV–Visible spectrophotometer. The equilibrium adsorption removal (E%) was calculated using the following equation:

$$E\ \% = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

3. Results and discussion

3.1. Characterization for adsorbent/adsorbate

FESEM Study on Activated Carbon before and after Adsorption

Field emission scanning electron microscopy (FESEM) of the surface morphology of biomass (coconut shells)-based activated carbon before the adsorption of dye was taken at a magnification of 10,000×, as shown in **Figure 2a**. A highly heterogeneous, porous structure was observed by micrography; the aspect ratio, irregularly fractured surface, sharp edge and well-defined macropores and micropores can all be identified. This morphology is characteristic of chemical activated carbon materials, where the dehydrating

agent (e.g., H_3PO_4) facilitates the creation of a porous skeleton and volatile species during carbonisation. The rough surface texture with numerous cavities in TEM images suggests that a large available surface area and a high density of active sites are present, which are essential characteristics for the effective adsorption of organic pollutants, such as MG. The open and interconnected pores are conducive to the diffusion of the adsorptive molecules into the inside of the adsorbent. Altogether, the FESEM photograph depicts a suitable surface superstructure for adsorption in wastewater treatment and environmental decontamination[16, 17].

The surface morphology of the activated carbon after adsorbing dye molecules (MG dye) was obtained from a field-emission scanning electron microscopy (FESEM) image at a magnification of 10,000×, as shown in **Figure 2b**. In contrast to tomography based on the pre-adsorption surface, the morphology is smoother and more compact, with fewer visible pores. Pore entrances and roughness observed before the adsorption process appear to be partly shielded by adsorbed material that creates an irregular film covering the surface. This observation indicates a strong connection between the dye molecule and the carbon surface. It confirms the possible occupation and even filling of the surface hollows and active sites of carbon[18]. The surface coverage with adsorbate of the dye is good evidence of the interaction that occurred between the activated carbon and the dye, thus confirming the adsorption property of the material. The surface topography modifications, partially pore plugging, as well as a decrease in roughness and the presence of adsorbed clusters/layers, are in agreement with the visual evidence of dye filling/coverage after adsorption, respectively. These results also demonstrate that activated carbon prepared from biomass can be successfully used to remove organic pollutants from aqueous solutions, indicating its high surface affinity and adsorption capabilities[19, 20].

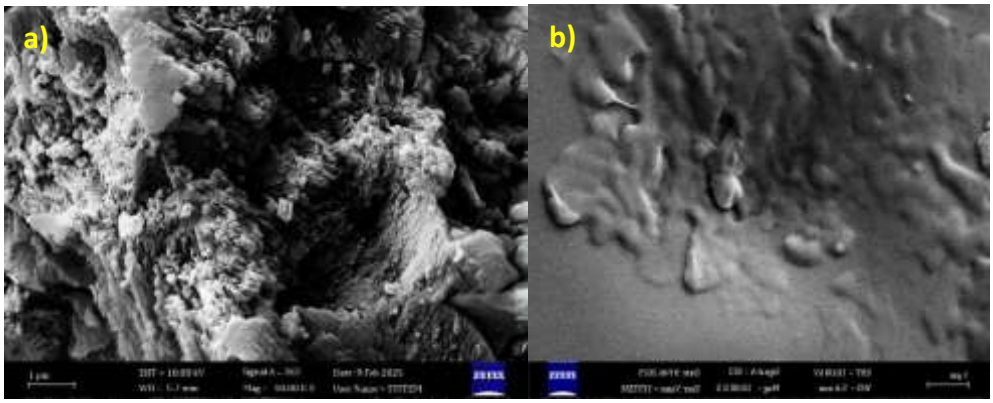

Figure 2: FESEM a) activated carbon before adsorption, b) after adsorption

TEM Investigations of Activated Carbon Prepared from Coconut Shell

The transmission electron microscopy (TEM) image represents the nanostructural morphology of coconut shell-based activated carbon, as shown in **Figure 3**. The particles appear to be somewhat irregular in shape and size, with the presence of evident nano-aggregates and nanoporosity[18]. The structures display a mix of dense and more transparent areas, which can be interpreted as fingerprints of compact carbon clusters and lighter, possibly porous or amorphous zones. Morphology indicated the chemical activation and an eventual partial graphitization, induced probably through phosphoric acid and high-temperature treatment, that allowed the capture of the calcium of the adsorbent in a disordered carbon matrix with nanometric porosity. The detected dark patches in the image signify higher electron density, possibly due to the denser forms of carbon nanodomains, whereas the bright sites may be indicative of pore walls or voids within the carbon skeleton[21, 22].

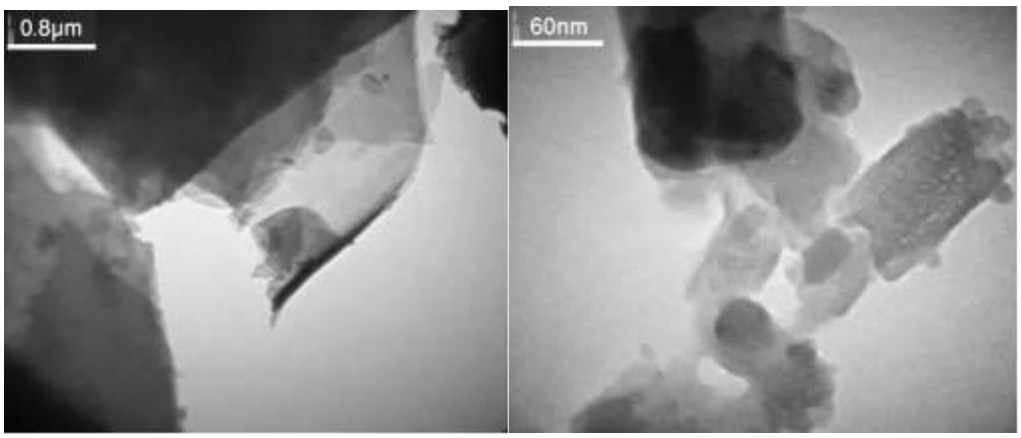

Figure 3. Transmission electron microscopy (TEM) image of activated carbon

3.2. Influence of H_3PO_4 on shell mass ratio of the activated efficiency

Figure 4 shows the effect of phosphoric acid-to-shell mass ratio on the per cent activation (E%) of the activated carbon prepared from coconut shell. Five ratios of mass were considered: 1:1, 1.5:1, 2:1, 2.5:1, and 3:1. Our calculations show that the activation effectiveness is strongly affected by the mass ratio of H_3PO_4 to shell. The retention efficiency was very low (about 10% at 1:1), indicating poor acid penetration and inadequate pore structure development. When the ratio was increased to 1.5:1, the activation efficiency rose to nearly 70%, indicating a more efficient chemical interaction between the acid and the carbon framework. The best yield (≈99%) was attained at a mass ratio of 2:1 and this seems to provide the best condition to obtain an active carbon having a high specific surface area (pore volume). Dry-loading with a higher acid amount of 2.5:1, however, slightly decreased the efficiency to 80%, which was probably due to partial loss of porosity or saturation. At the highest ratio (3:1), the efficiency was further reduced by approximately 60%, which can be attributed to the abundance of acid, which etches or blocks the porous structures. These results indicate that: (1) although phosphoric acid is essential for activation, it must be at an optimum ratio (2:1) that promotes adequate chemical activation but without loss of the structure. It is detrimental to the quality of the activated carbon if the acid treatment extends beyond this point [4, 13].

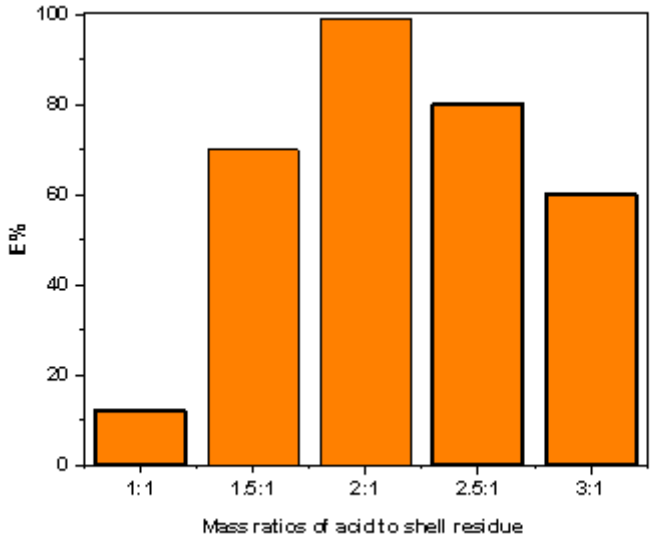

Figure 4: Effect of H_3PO_4 on shell mass ratio on activated efficiency

3.3. Influence of calcination temperature on the activation activity

A similar trend is depicted in the bar graph (**Figure 5**), where different temperatures (200°C to 600°C) were tested to determine the optimal fit for maximum activation. There was a relatively low activation efficiency of approximately 45% at 200°C, indicating that the temperature did not provide sufficient energy to develop porosity or activate surface sites. Upon increasing the temperature to 300°C, a reasonable efficiency ~of 70% indicated a better decomposition of volatile matter and the beginning of workworthy pore production[23]. Efficiency further increased to around 80% at 400 °C, indicating that the growth of micro- and mesoporous structures was enhanced. The best activation efficiency (nearly 99%) was obtained at 500°C, suggesting it as the optimal calcination temperature for producing a highly porous and adsorbent active carbon. A slight decrease in performance to 90% was observed at 600 °C, which can be attributed to structural deformation or excessive carbon burn-off, resulting in shrinkage of the pore structure and loss of surface area available for adsorption. It has been proven that the calcination temperature is a key factor in the activation process. And though typically activation is facilitated by relatively higher temperatures, a specific befalling limit – such as 500°C here – may apply where any higher temperature might actually reduce the adsorption potential of the material[24].

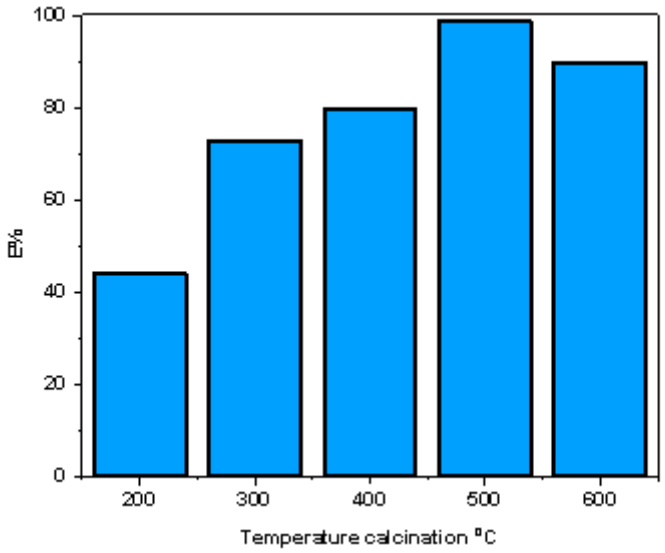

Figure 5. Effect of calcination temperature on the activation activity

3.4. Impact of the dosage of activated carbon on the efficiencies of dye removal

Figure 6 shows the adsorbent dosage (g) of the activated carbon against the MG dye E% in an aqueous solution. With the dosage increasing from 0.02 g to 0.08 g, the removal performance can be significantly improved, indicating that the amount of adsorbent plays a crucial role in the adsorption process. Removal efficiency is approximately 70% at a concentration of 0.02 g. Increasing the dosage to 0.03 g increases the efficiency to 80%. When the amount of AC loaded reaches 0.04 and 0.05 g, the removal efficiencies reach 83% and 90%, respectively. At 0.06 g, efficiency reaches approximately 94%, and at 0.08 g, it goes a maximum of 99%. The increase is due to the rise in the number of active sites and the available surface area for adsorption as more activated carbon was added. The increased surface area leads to enhanced interaction between the dye molecules and the adsorbing agent, resulting in improved adsorption[25]. However, although not fully utilised yet, one can already infer that the rise in efficiency rate starts to flatten at higher doses, meaning that above a specific value, the system is no longer functioning at its maximum adsorption, indicating that the system is near or at saturation. Beyond certain weights of adsorbent, however, little efficiency was gained, possibly as a result of a lack of optimum concentration of the dye in the solution and particle aggregation that results in the loss of the effective surface area. In general, these findings reveal that

the optimal dosage of an adsorbent is a key factor in achieving high removal efficiency while maintaining the economic and material effectiveness of the AC in wastewater treatment[26].

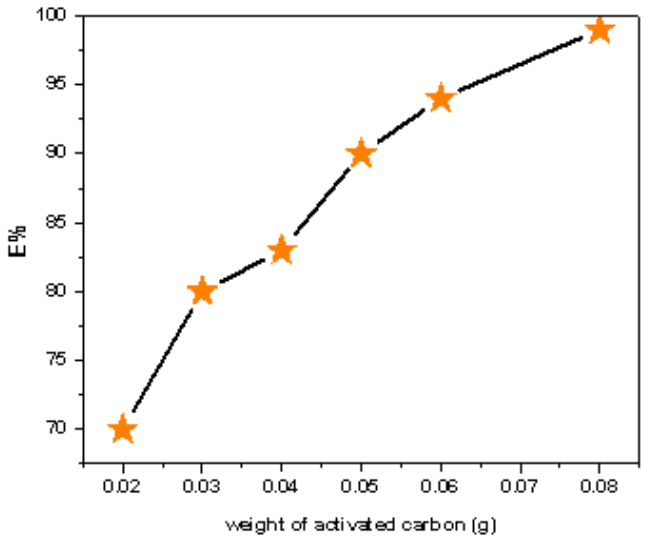

Figure 6. Effect of the weight of activated carbon on the removal of MG dye

3.5. Impact of pH on MG dye adsorption by activated carbon

The plot represents the effect of solution pH on the removal percentage (%) of MG dye using activated carbon as an adsorbent. The investigated pH range is 2-11, and a clear trend in MG adsorption is observed. MG at pH 2 had a removal efficiency of more than 20%, which indicates poor adsorption in highly acidic conditions. When the pH level rises to 4 and 6, efficiency is significantly improved, reaching approximately 55% and 60%, respectively. It suddenly and rapidly increases at pH 7, reaching almost 90% of its maximum efficiency. The uptake of lead increases slightly with pH (9 and 10, reaching a maximum of 93% and 99%)[27, 28]. This phenomenon can be attributed to the ionization property of the cationic dye (MG) and the surface charge property of the activated carbon. At low pH, the carbon surface is protonated, imparting a positive charge that repels the anionic dye, resulting in lower adsorption. However, with increasing pH, the carbon surface becomes negatively charged, which increases the electrostatic attractive force between the dye molecules and the adsorbent surface, resulting in a higher removal efficiency. The results clearly verify that the adsorption of MR in activated carbon is enhanced by the application of an alkaline system, which is highly pH-dependent, and the process's performance is superior under a basic medium (pH ≥ 10)[7, 29].As shown in **Figure 7**.

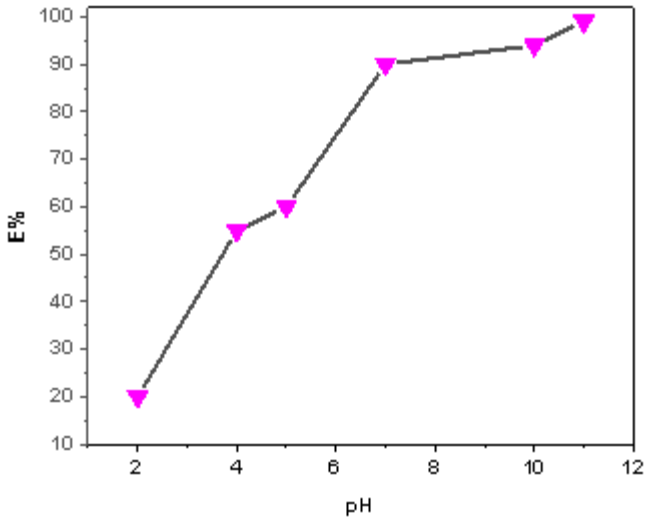

Figure 7. Effect of pH on MG dye adsorption by activated carbon

3.6. The effect of equilibrium time

The impact of equilibrium time on the adsorption of MG dye was studied. The experimental results are shown in **Figure 8**. A time of 60 minutes was used as the basic experimental parameter to ensure that all active sites reached equilibrium. It was observed that as time increased, the removal rate increased rapidly during the first 20 minutes. However, as more active sites became available, the rate of removal slowed until equilibrium was reached. Expanding the time did not significantly improve the dye removal process. As the adsorption time increased, it became more challenging to fill all the active sites, resulting in a slower removal process until equilibrium was reached. Therefore, the closer the adsorption process approached equilibrium per hour at a given MG dye concentration, the greater the rate of removal. This was of great importance for the removal of MG dye from wastewater[3].

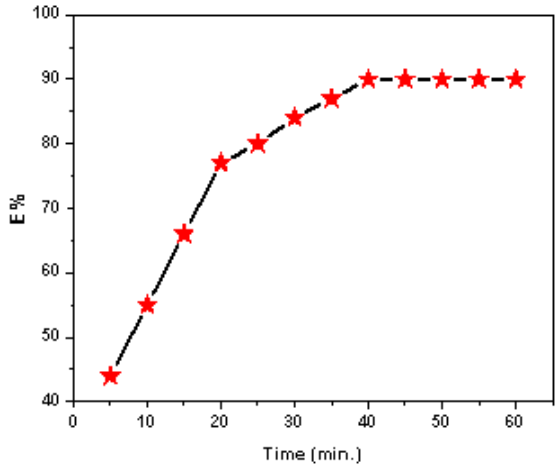

Figure 8. The effect of equilibrium time on the removal of MG dye

3.7. Effect of temperature

One of the most critical factors affecting the adsorption process is temperature, which is considered a fundamental factor. It is regarded as a crucial aspect that must be considered in the removal of hazardous pollutants, including dyes, as it determines whether the adsorption process is endothermic or exothermic. In this study, the effect of temperature on the removal rate of MG dye using acid-activated carbon was investigated. As shown in **Figure 9**, the results indicate that the removal rate increases significantly with increasing temperature. This is attributed to the increased mobility of the dye molecules, which helps increase the dye's diffusion from the aqueous solution to the surface of the activated carbon. This increased mobility subsequently allows for more active sites on the surface of the activated carbon and dye molecules, leading to increased removal efficiency[30].

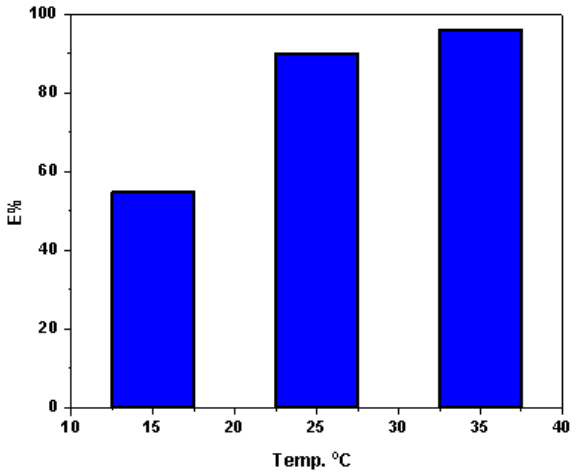

Figure 9. Effect of temperature on the removal of MG dye

3.8. Estimates of surface regeneration with varying regenerating agents

The effect of the three regeneration agents - HCl, NaOH, and H_2O - on the regeneration efficiency (E%) of the activated carbon after dye adsorption is shown in Figure 10. The acidic medium HCl showed the best dye removal regeneration with a regeneration efficiency ~99%, which means it is very effective to desorb the trapped dye molecules and to restore the carbon adsorption capacity. The strong acidic condition may favour the destruction of electrostatic forces between the adsorbed dye (MG) and the surface functional group on the carbon material[31].The regeneration efficiency was 45% by using NaOH (an alkali solution). Although basic conditions can promote the desorption of some anionic dye molecules, they can also modify or eliminate functional groups on the activated carbon surface, which may decrease the accessibility of active sites.The lowest % regeneration was obtained with the neutral medium water – which is less effective, causing a separation efficiency of only ~10%. This lines up with being unable to separate the dye molecule from the carbon surface in a water solution without a stimulating chemical reagent. The results reveal that the acidic regeneration process, especially with HCl solution, is the best to regenerate the adsorption capacities of castor leaf-based AC. It is a tangible and efficient way of increasing the lifetime and reuse of adsorbents in wastewater treatment[32].

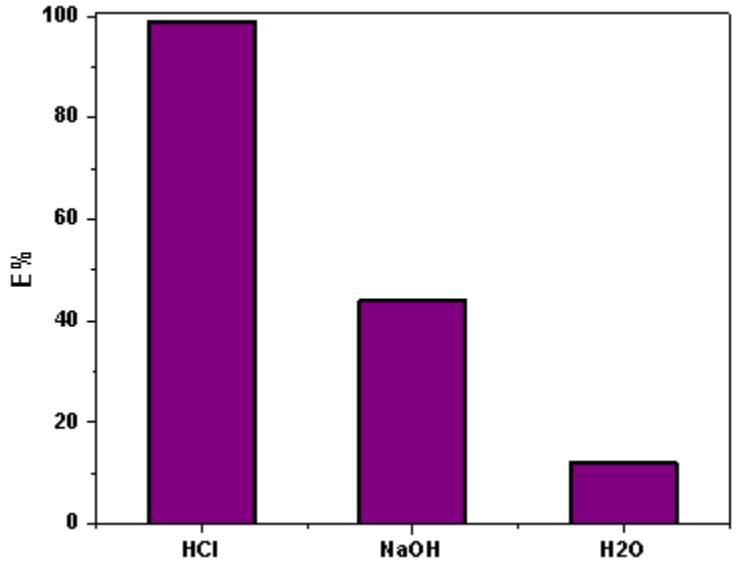

Figure 10. Estimates of surface regeneration with varying regenerating agents

3.9. Influence of regeneration cycles on adsorption performance

The bar chart illustrates the impact of repeated regenerations on the adsorption efficiency (E%) of castor leaf activated carbon. Number of regeneration cycles (0, initial use, 0+1, 0+1+2, and 0+1+2+3+4) as shown in **Figure 11**. The results show a gradual decrease in adsorption capacity during repeat cycles: In the case of fresh adsorbents (at Cycle 0), the adsorption efficiency was around 98%, demonstrating the high initial capacity of activated carbon for dye molecule adsorption (e.g MG dye)[33]. The efficiency was slightly reduced to approximately 93% after the initial regeneration cycle, indicating good retention of the adsorption capacity. The efficacy dropped to 89% and 87% by Cycle 2 and 3, respectively. After four cycles, the adsorbent retained approximately 80% of its initial efficiency, indicating a modest reduction in efficiency due to partial pore clogging, surface fouling, or slow decay of the active centres. This efficiency is high enough for its regeneration by reusing four times the developed activated carbon, ensuring good reusability, which is well-suited to the regeneration situation. The results of these studies have suggested that castor leaf-based activated carbon is an environmentally friendly and regenerable adsorbent, with potential for use in multi-cycle applications for dye removal in wastewater treatment industries[34].

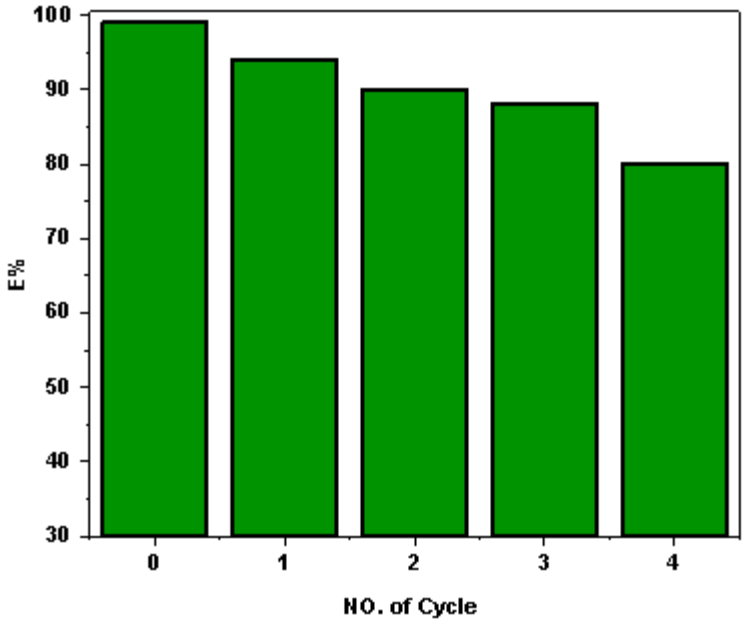

Figure 11. Influence of regeneration cycles on adsorption performance

3.10. Mechanism adsorption

Adsorption of Malachite Green (MG) cationic dye on phosphoric acid-activated carbon: several possible interaction mechanisms. Primarily, the electrostatic attraction can be an important factor, because the majority of the surface of the activated carbon is negatively charged, including with functional groups such as –OH, –COOH, or $–PO_4^{3-}$, which can attract very well positively charged dye molecules, especially near neutral or basic pH. Moreover, π–π interaction could happen between the aromatic rings of MG and the graphitic domains on the surface of AC, thus favoring the adsorption. Hydrogen bonding between functional groups on the carbon surface and N- or O-atoms in dye structure can also be responsible. A significant enhancement effect of dye removal efficiency was achieved with rising temperatures, which indicates an endothermic nature of the adsorption process, and chemisorption as primary mechanism. The aggregate interaction could lead to the increased adsorption capacity and efficiency as observed in the present work[35].

4. Conclusion

In this work, coconut shell waste was successfully converted into activated carbon using chemical activation with phosphoric acid (H_3PO_4) at different acid ratios. The optimal activation condition was achieved at a 2:1 ratio, which resulted in the highest removal capacity. Surface characterisations, such as TEM and FESEM images, showed that the prepared ACs had a high specific surface area, abundant micro and mesoporous structure, and heterogeneous rough surface morphology, which is advantageous for adsorption. Adsorption properties. The adsorption capacity of the material toward the MG dye was excellent, achieving a removal efficiency of nearly 90% under optimal conditions. The adsorption efficiency was dependent on the process parameters, viz., pH, adsorbent dosage, and contact time, with maximum dye removal occurring in alkaline medium due to increased electrostatic attraction between negatively charged carbon surface and dye anions. Additionally, the regenerated tests demonstrated that the activated carbon retained its structural integrity and adsorption capability after multiple adsorption–desorption cycles. The highest Regeneration efficiency (92-99% of initial sorption capacity) was recorded for hydrochloric acid among the screened regeneration agents. This study provides a new insight into the promising utilisation of phosphoric acid-activated coconut shell as a green-sourced, efficient, and reusable adsorbent for the elimination of synthetic dyes from wastewater, which could be conducive to designing eco-friendly and low-cost water treatment techniques.

Conflict of interest

The authors declare there is no conflict of interest.

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