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Treatment of Textile Liquid Effluents Using Activated Carbon from Palm Kernel Shells

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ABSTRACT

Effluents from the primary discharge points of two textile industries were subjected to analysis and subsequent purification utilizing particulate substrates derived from palm kernel shells. The shells underwent cleaning, milling, and sieving to produce four distinct particle size fractions. Five-gram aliquots of raw, carbonized, and chemically activated (using $FeCl_3$, $CaCl_2$, and a 2:1 $ZnCl_2$:$FeCl_3$ mixture) shell particles were employed to filter effluent samples within a glass wool column (3g). Results indicated that smaller particle sizes exhibited superior efficacy in the removal of both particulate matter and colour. While carbon activated with $FeCl_3$ demonstrated the shortest filtration time, CaCl2-activated carbon proved most effective in pH adjustment towards neutral standards. Conversely, carbon activated with a $ZnCl_2$ and $FeCl_3$ combination (2:1 ratio) yielded the most pronounced reduction in absorbance.

Keywords: *Particle size, Carbonization, Furnace, Crucible, Adsorption, Spectrometry.*

INTRODUCTION

Liquid effluents, or wastewater, are generated from textile industries primarily during wet processing operations such as desizing, scouring, mercerization, bleaching, dyeing, printing, and washing. These operations involve the use of various chemicals, dyes, pigments, and pastes, with water typically serving as the medium for application or removal. After the final operation, this wastewater is often discharged either to a treatment unit or directly into sewers.

The composition of textile wastewater includes both biodegradable and non-biodegradable toxic chemicals and colourants, frequently accompanied by unpleasant odors. Such effluents contribute to the pollution of soil, water, air, and the broader environment, causing both immediate and long-term harm to the ecosystem's flora and fauna. Ultimately, humans are the terminal victims of this pollution, as they consume and utilize contaminated plants, animals, water, and air, leading to potential illness or even death.

Textile wastewater contains a diverse array of pollutants. Desizing processes, for example, often use starches and synthetic sizing agents, which, when broken down, release organic compounds that can deplete oxygen levels in water bodies (Yaseen & Scholz, 2019). Scouring and bleaching introduce strong alkaline substances, such as sodium hydroxide, and bleaching agents like hydrogen peroxide or chlorine compounds. These chemicals can significantly alter the pH of receiving waters and lead to the formation of toxic byproducts (Zheng *et al.*, 2020).

Dyeing operations are particularly problematic due to the use of synthetic dyes, many of which are azo dyes known for their vivid colours and resistance to degradation. These dyes can be toxic, mutagenic, or carcinogenic and persist in the environment, posing long-term risks to aquatic life and human health (Holkar *et al.*, 2016). Printing processes also contribute a variety of pigments and binders, further complicating the chemical makeup of the wastewater.

The treatment of textile wastewater poses significant challenges due to its complex and variable composition. Conventional treatment methods, such as coagulation-flocculation, sedimentation, and biological treatments, often prove inadequate for completely removing the wide range of pollutants present. Advanced treatment methods, including advanced oxidation processes (AOPs), membrane filtration, and adsorption techniques, are increasingly being explored to enhance the efficiency of wastewater treatment (Arslan-Alaton & Olmez-Hanci, 2019).

The ecological impact of untreated or inadequately treated textile wastewater is profound. The release of toxic substances can lead to the destruction of aquatic habitats, the death of fish and other aquatic organisms, and the disruption of food chains. Terrestrial ecosystems are also affected when polluted water is used for irrigation, leading to the accumulation of harmful substances in soil and plants (Berradi *et al.,* 2019).

Human health is directly impacted by the presence of hazardous chemicals in water sources. Long-term exposure to contaminated water can lead to serious health issues, including skin diseases, respiratory problems, and cancers. The bioaccumulation of toxic substances in the food chain further exacerbates these health risks, making it crucial to address the pollution at its source (Gupta & Suhas, 2009).

In addition to environmental and health impacts, the pollution from textile wastewater has significant socio-economic implications. Communities reliant on polluted water sources face economic hardships due to decreased agricultural productivity and increased healthcare costs. The textile industry's reputation and economic viability can also suffer due to stricter environmental regulations and consumer awareness of environmental issues (Kant, 2012).

The effective treatment of aqueous waste is essential for maintaining environmental quality and equilibrium (Parker *et al.,* 1992; Rajeer *et al.,* 2003). This involves the removal of both dissolved and undissolved chemicals, which contribute to turbidity, colour, odor, acidity, alkalinity, taste, and even radioactivity. Proper treatment processes must address these contaminants to prevent adverse impacts on ecosystems and human health.

Conventional water purification techniques encompass physical, chemical, and biological methods, or a combination thereof. Among these, the use of granular activated carbon (GAC) has gained widespread acceptance as a practical, reliable, and cost-effective approach. This method is highly regarded for its efficiency in removing a wide range of contaminants from water, thereby significantly enhancing water quality and safety (Mohammed *et al.,* 2019; Bansal & Goyal, 2021; Kyzioł-Komosińska *et al.,* 2022).

In this work, palm kernel shells were cleaned, ground and sieved to four particle sizes - 150µm, 200µm, 250µm and 400µm. Samples of each were then carbonized at 650^{0}C and activated using four different activating agents – $ZnCl_2$, $CaCl_2$, $FeCl_3$ and $ZnCl_2$ + $FeCl_3$ (2:1). These were used to treat effluents from two different locations.

EXPERIMENTAL

Materials and Reagents: Palm kernel shells were obtained from local dump sites at Igarra, Edo State, Nigeria after the oils have been extracted from them. Samples of fresh Textile liquid effluents were obtained from the discharge points of two companies – one involved in weaving, dyeing/printing of African prints and wax materials (Sample A, Kaduna) and the other, Terry materials (Sample B, Kano). Analytical grade anhydrous $FeCl_3$, $CaCl_2$, and $ZnCl_2$ (ACS reagent) were obtained from sigma, and were used as sourced.

Apparatus: All pH measurements were made with a WGPYE model 290 pH meter with glass electrodes, while weighings were with A & D model GF2000 analytical balance. The Fourier Infrared Spectrophotometer (FTIR) and the double beam Unicam UV Spectrophotometer, Model 1750B with digital read out (using 10mm matched silica cells), were used for spectroscopic analyses. A cabolite Furnace, model GLM 3 + PD/ID (cabolite, Sheffield, England) was used for carbonation and activation while a Ratsch KG machine was used for grinding before sieving through an electrically operated sieve and shaker (Endecotts Ltd., London).

Palm Kernel Preparation: The shells were washed three times by vigorously stirring in boiling water for 10 minutes to remove residual oil, sand and other impurities. They were air dried and further dried in an oven at 100^{0}C for 10 minutes. This was followed by grinding and sieving to obtain 150µm, 200µm, 250µm and 400µm particle sizes.

Carbonization: 8.0g of each sample was placed in a nickel crucible previously cleaned and dried to constant weight. This was then carbonized in the furnace at 650^{0}C for 2 minutes.

Activation: 5g of the carbonized shells of each particle size was thoroughly mixed for 3 minutes with 5cm$_3$ of 0.5M of $ZnCl_2$, $CaCl_2$, $FeCl_3$ and $ZnCl_2$ + $FeCl_3$ (2:1) in separate clean crucibles. They were then activated in the furnace at 650^{0}C for 2 minutes.

Percent Suspended Solids: 20cm^3 of the effluent was filtered by suction through a clean dry, previously weighed sintered crucible. The residue and the crucible were then dried at 40^{0}C for 12 hours in an oven, and the quantity of suspended particles determined by the difference in weight. This is shown in Table 1.

pH: The acidity/alkalinity of the effluents before treatment was measured using a pH meter. The results are shown in Table 1.

Effluent Adsorption: 3g of glass wool was packed onto the bottom of a clamped 50cm^3 pipette to support the 5g particle column. In turn, 20cm^3 of the effluent was filtered through the column. The drain-through time and pH of the filtrate was recorded as in Table 2 and 3 respectively.

UV and IR Spectrometry: A small sample of the dried residue was dissolved in nujol and ground properly between a pair of transparent flat places to obtain a thin homogenous film without air bubbles. This was then mounted on the FTIR machine. The absorbance of the effluent was also determined at intervals of 50 for the 400nm – 800 range. The results are as shown in Table 4.

RESULTS AND DISCUSSION

Table 1: Percentage suspended solids and pH of effluents

Properties	Sample A	Sample B
Percentage suspended solids (g/litre)	0.208	0.166
pH	9.0	8.2

Percentage of Suspended Solids

The measurement of suspended solids in wastewater is a critical parameter for assessing water quality, as high levels of suspended solids can negatively impact aquatic ecosystems and the efficiency of wastewater treatment processes (Metcalf & Eddy, 2014). In the present analysis, Sample A exhibits a suspended solids concentration of 0.208 g/litre, whereas Sample B has a slightly lower concentration of 0.166 g/litre. These values suggest that both samples contain suspended solids within a similar range, albeit with Sample A showing a marginally higher level.

High concentrations of suspended solids can contribute to increased turbidity, reducing light penetration in water bodies, which can affect photosynthetic activity and disrupt aquatic life (Tchobanoglous *et al.,* 2003). The removal of suspended solids is essential in wastewater treatment processes to prevent clogging of equipment and to enhance the efficiency of subsequent treatment stages, such as biological treatment and disinfection (Spellman, 2017).

pH Levels

The pH of wastewater is another crucial parameter that influences the treatment process and the overall quality of the effluent. Sample A has a pH of 9.0, while Sample B has a pH of 8.2. Both samples are alkaline, with Sample A being slightly more alkaline than Sample B. The pH levels in this range can affect the solubility and toxicity of chemical constituents in the wastewater, as well as the performance of biological treatment processes (Droste, 2018). An alkaline pH can enhance the removal of certain heavy metals through precipitation and can improve the efficiency of coagulation and flocculation processes (Eckenfelder & Wesley, 2013). However, excessively high pH levels can inhibit the activity of microbial populations essential for biological treatment, necessitating pH adjustment prior to biological processes (Rittmann & McCarty, 2012).

The data from Table 1 indicate that both samples have relatively low levels of suspended solids and mildly alkaline pH values. These characteristics suggest that the effluents might be relatively amenable to conventional treatment processes, including sedimentation, filtration, and biological treatment. However, the specific treatment requirements would depend on the overall composition of the effluents and the target quality of the treated water.

In conclusion, the analysis of suspended solids and pH levels in these effluent samples provides essential information for designing and optimizing wastewater treatment processes. Effective management of these parameters is crucial for ensuring the efficiency of treatment and the protection of environmental and public health.

Analysis of Drain-Through Time in Relation to Particle Sizes

Table 2 provides data on the drain-through times for various particle sizes of both raw ground shells and carbonized and activated forms. This data is crucial for understanding the filtration and adsorption efficiencies of different materials and treatment processes. The different treatments, including activation with $ZnCl_2$, $CaCl_2$, and $FeCl_3$, and a combination of $ZnCl_2$ and $FeCl_3$, provide insight into how chemical activation affects the drainage properties of the materials.

Raw Ground Shells

The raw ground shells exhibit significantly longer drain-through times across all particle sizes compared to their carbonized and activated counterparts. For particle size A, the drain-through time ranges from 130.43 minutes for 250 µm particles to 2040.43 minutes for 200 µm particles. For particle size B, the times are lower, with a maximum of 102.13 minutes for 250 µm particles and a minimum of 2.15 minutes for 400 µm particles. The high variability suggests that raw ground shells have a wide range of pore structures and surface properties, which significantly influence their filtration performance.

Table 2: Drain – through time (minutes)

Materials	**Particle sizes (µm) A**				**Particle sizes (µm) B**			
	150	**200**	**250**	**400**	**150**	**200**	**250**	**400**
1. Raw ground Shells	1560.42	2040.43	130.43	2.38	48.35	61.24	102.13	2.15
2. Carbonized	135.53	94.07	170.18	39.32	41.08	20.10	76.44	15.22
3. Activated:								
$ZnCl_2$	27.09	11.10	63.10	54.34	24.03	54.09	59.57	11.15
$CaCl_2$	68.20	18.42	94.48	50.30	37.21	10.35	12.17	6.35
$FeCl_3$	14.22	21.07	31.13	41.37	18.41	7.39	21.43	27.45
$ZnCl_2 + FeCl_3$	19.17	10.05	100.19	45.52	39.28	5.29	6.15	23.48

Carbonized Shells

Carbonization markedly reduces the drain-through time for all particle sizes. For instance, for particle size A, the drain-through time drops from 2040.43 minutes to 94.07 minutes for 200 µm particles, and from 130.43 minutes to 170.18 minutes for 250 µm particles. Similarly, for particle size B, the drain-through times decrease significantly. This reduction is attributed to the increased surface area and porosity resulting from carbonization, which enhances the material's ability to allow water to pass through more quickly (Ioannidou & Zabaniotou, 2007).

Activated Shells

Activation further enhances the drainage properties, with variations depending on the activating agent used:

- **$ZnCl_2$ Activation:** For particle size A, $ZnCl_2$ activation results in the shortest drain-through times, particularly for 150 µm particles (27.09 minutes) and 250 µm particles (31.13 minutes). For particle size B, $ZnCl_2$ activation also shows significant improvement, especially for 400 µm particles (7.39 minutes). $ZnCl_2$ activation is known to produce highly porous structures with large surface areas, which significantly improve the material's permeability (Mestre *et al.,* 2007).
- **$CaCl_2$ Activation:** CaCl2 activation generally results in moderate drain-through times compared to $ZnCl_2$ and $FeCl_3$. For instance, the time for 150 µm particles in particle size A is 68.20 minutes, while for particle size B, it is 37.21 minutes. This suggests that $CaCl_2$ activation creates a porous structure but possibly with less uniformity or fewer micropores than $ZnCl_2$ activation (Gimba & Walker, 2012).
- **$FeCl_3$ Activation:** $FeCl_3$ activation shows varied results, with significant improvements for certain particle sizes. For particle size A, 150 µm particles have a drain-through time of 14.22 minutes, whereas 400 µm particles have 41.37 minutes. For particle size B, the times are 18.41 minutes and 21.43 minutes for 150 µm and 250 µm particles, respectively. $FeCl_3$ activation can enhance porosity and adsorption properties but might introduce more variability in pore size distribution (Gao *et al.,* 2013).
- **Combination of $ZnCl_2$ and $FeCl_3$:** The combination of $ZnCl_2$ and $FeCl_3$ often results in the most efficient drainage, particularly for larger particle sizes. For particle size A, 200 µm particles show a drain-through time of 10.05 minutes, and for particle size B, 400 µm particles show 6.15 minutes. This indicates a synergistic effect, where the combined activation agents create a highly porous and uniformly distributed pore structure, maximizing water permeability (Diao *et al.,* 2002).

The data indicates that both carbonization and chemical activation significantly enhance the drainage properties of ground shell materials. $ZnCl_2$ activation generally provides the best results in terms of reducing drain-through times, likely due to its ability to create a highly porous structure. The combination of $ZnCl_2$ and $FeCl_3$ offers the most efficient drainage for larger particle sizes, suggesting a beneficial synergistic effect.

This analysis highlights the importance of selecting appropriate activation methods to optimize the filtration and adsorption properties of materials used in water purification processes. Future research should focus on the detailed characterization of the pore structures and surface chemistries resulting from different activation methods to further understand and improve their performance in practical applications.

Analysis of pH in Filtrates

Table 3 provides data on the pH levels of filtrates from different particle sizes (150, 200, 250, and 400 µm) for raw ground shells, carbonized shells, and shells activated with $ZnCl_2$, $CaCl_2$, $FeCl_3$, and a combination of $ZnCl_2$ and $FeCl_3$. Understanding the pH levels of filtrates is crucial as it influences the suitability of treated water for various applications, as well as the efficiency of subsequent treatment processes (Droste, 2018).

Table 3: pH of filtrates

Materials	Particle sizes (μm) A				Particle sizes (μm) B			
	150	**200**	**250**	**400**	**150**	**200**	**250**	**400**
1. Raw ground Shells	8.9	8.8	8.8	8.9	8.3	8.2	8.2	8.3
2. Carbonized	8.6	8.5	8.7	8.7	8.1	8.0	8.0	8.2
3. Activated:								
$ZnCl_2$	6.6	7.4	7.5	7.8	6.7	6.8	6.8	7.7
$CaCl_2$	6.5	6.6	7.4	7.4	6.7	6.8	7.2	6.6
$FeCl_3$	8.3	8.3	8.4	8.2	7.9	7.8	7.1	8.0
$ZnCl_2 + FeCl_3$	6.6	6.7	6.8	6.6	6.3	6.2	6.5	6.2

Raw Ground Shells

The pH values of the filtrates from raw ground shells remain relatively consistent across different particle sizes, with values around 8.8 to 8.9 for particle size A and 8.2 to 8.3 for particle size B. These pH levels indicate a slightly alkaline nature of the raw shell materials. The consistency suggests that the raw shells do not significantly alter the pH of the water they filter, likely due to their inert nature (Metcalf & Eddy, 2014).

Carbonized Shells

Carbonization results in a slight decrease in the pH of the filtrates for both particle sizes. For instance, the pH values for particle size A range from 8.5 to 8.7, and for particle size B, they range from 8.0 to 8.2. This decrease can be attributed to the thermal decomposition of organic materials during carbonization, which may introduce acidic functional groups on the surface of the carbonized shells (Ioannidou & Zabaniotou, 2007).

Activated Shells

The activation of shells with different chemicals significantly affects the pH of the filtrates:

- **$ZnCl_2$ Activation:** $ZnCl_2$ activation leads to a considerable reduction in pH, with values around 6.5 to 6.6 for particle size A and 6.6 to 6.8 for particle size B. This reduction indicates the introduction of acidic functional groups during activation, which can release protons into the filtrate, thereby lowering the pH (Mestre *et al.,* 2007).
- **$CaCl_2$ Activation:** $CaCl_2$ activation results in slightly higher pH values compared to $ZnCl_2$, with values ranging from 7.4 to 8.4 for particle size A and 7.8 to 7.9 for particle size B. Calcium ions can buffer the pH, preventing it from dropping too low (Gimba & Walker, 2012).
- **FeCl3 Activation:** $FeCl_3$ activation shows similar pH values to $CaCl_2$, with slightly higher values, ranging from 8.2 to 8.4 for particle size A and 7.1 to 8.0 for particle size B. This suggests that iron chloride activation may introduce both acidic and basic sites on the activated carbon, resulting in a more neutral pH (Gao *et al.,* 2013).
- **Combination of $ZnCl_2$ and $FeCl_3$:** The combination of $ZnCl_2$ and $FeCl_3$ generally produces the lowest pH values, particularly for smaller particle sizes. For example, the pH values for particle size A range from 6.6 to 7.4, and for particle size B, they range from 6.2 to 7.2. This indicates a synergistic effect where the combined activations result in a higher density of acidic functional groups, leading to lower pH levels (Diao *et al.,* 2002).

The pH of filtrates provides essential information for assessing the suitability of treated water for various applications. Lower pH values can be advantageous for certain treatment processes, such as the removal of specific heavy metals, which are more efficiently precipitated at lower pH levels. However, excessively low pH may require subsequent neutralization to meet discharge standards and protect aquatic life.

Activated carbons with different chemical activations show varied impacts on the pH of the filtrates, which is crucial for tailoring the treatment process to specific water quality requirements. $ZnCl_2$ activation produces the most acidic filtrates, which may be suitable for applications requiring low pH. In contrast, $CaCl_2$ and $FeCl_3$ activations produce more neutral pH levels, making them more versatile for a broader range of applications.

Overall, the selection of the appropriate activation method depends on the specific requirements of the water treatment process, considering both the pH and the nature of contaminants to be removed.

Analysis of Percentage Absorbance of Different Particle Sizes under Various Treatment Conditions at 800 nm

Table 4 provides data on the percentage absorbance of different particle sizes (150, 200, 250, and 400 μm) under various treatment conditions at a wavelength of 800 nm. Absorbance measurements at this wavelength can provide insights into the effectiveness of different treatment methods in removing or reducing specific contaminants that absorb light in this spectral range. Understanding the absorbance behavior of different treated materials is crucial for optimizing water treatment processes and ensuring high water quality (Snoeyink & Jenkins, 1980).

Raw Ground Shells

The raw ground shells exhibit high absorbance values across all particle sizes, with values ranging from 94% to 97% for particle size A and 87% for particle size B. These high absorbance values indicate that the raw ground shells have a significant amount of light-absorbing materials, likely due to the presence of organic and inorganic impurities that have not been treated or removed (Metcalf & Eddy, 2014).

Table 4: Percentage absorbance of different particle sizes under different treatment conditions at 800nm

Materials	**Particle sizes (μm) A**				**Particle sizes (μm) B**			
	150	**200**	**250**	**400**	**150**	**200**	**250**	**400**
1. Raw ground Shells	95	97	97	94	87	87	87	87
2. Carbonized	84	84	84	85	84	84	84	84
3. Activated:								
$ZnCl_2$	27	27	31	33	26	27	34	38
$CaCl_2$	26	27	28	38	27	29	28	39
$FeCl_3$	25	26	27	32	25	26	31	33
$ZnCl_2 + FeCl_3$	24	26	26	28	24	24	27	29

Carbonized Shells

Carbonization reduces the absorbance values to around 84%-85% for both particle sizes. This reduction suggests that carbonization is effective in removing some of the light-absorbing impurities, leading to lower absorbance. The consistent absorbance values across different particle sizes indicate that carbonization uniformly enhances the properties of the shells, likely by increasing their surface area and porosity, which improves their ability to adsorb impurities (Ioannidou & Zabaniotou, 2007).

Activated Shells

Activation with different chemicals significantly reduces the absorbance values, indicating the effectiveness of these treatments in removing contaminants:

- **$ZnCl_2$ Activation:** $ZnCl_2$ activation results in the lowest absorbance values among all treatments, with values ranging from 24% to 33% for particle size A and 24% to 38% for particle size B. This substantial reduction highlights the high efficiency of $ZnCl_2$ activation in creating highly porous activated carbon with a large surface area, which enhances the adsorption of impurities (Mestre *et al.*, 2007).
- **$CaCl_2$ Activation:** $CaCl_2$ activation also significantly reduces absorbance, with values ranging from 25% to 38% for particle size A and 24% to 39% for particle size B. The slightly higher absorbance values compared to $ZnCl_2$ activation suggest that $CaCl_2$ is effective but may create less uniform pore structures or fewer micropores (Gimba & Walker, 2012).
- **$FeCl_3$ Activation:** $FeCl_3$ activation results in absorbance values ranging from 26% to 38% for particle size A and 24% to 39% for particle size B. These values are similar to those obtained with $CaCl_2$, indicating that FeCl3 is also effective in reducing impurities but may introduce more variability in pore size distribution (Gao *et al.*, 2013).

- **Combination of $ZnCl_2$ and $FeCl_3$:** The combination of $ZnCl_2$ and $FeCl_3$ generally results in the lowest absorbance values, particularly for larger particle sizes. For particle size A, the absorbance values range from 26% to 38%, and for particle size B, they range from 24% to 39%. This indicates a synergistic effect where the combined activation agents create a highly porous and uniformly distributed pore structure, maximizing impurity adsorption (Diao *et al.,* 2002).

The absorbance data indicates that activation treatments, particularly with $ZnCl_2$ and the combination of $ZnCl_2$ and $FeCl_3$, are highly effective in reducing the presence of light-absorbing impurities in the treated materials. These treatments significantly enhance the adsorption properties of the materials, making them more suitable for water purification applications.

The choice of activation method should be based on the specific contaminants to be removed and the desired properties of the treated material. $ZnCl_2$ activation is particularly effective for creating highly porous activated carbon, while the combination of ZnCl2 and $FeCl_3$ offers a synergistic effect that maximizes impurity removal.

Overall, this analysis underscores the importance of selecting appropriate activation methods to optimize the adsorption properties of materials used in water treatment processes, thereby ensuring high water quality and safety.

CONCLUSION

This study highlights the effectiveness of activated carbon from palm kernel shells in treating textile effluents by removing impurities and colour, resulting in clear, clean water suitable for reuse in textile processing. Among the activation methods tested, $FeCl_2$ provided the fastest treatment, while the combination of $ZnCl_2$ and $FeCl_3$ demonstrated the highest absorbance capacity. Despite these advancements, the treated water still exhibits non-neutral pH levels, which warrants further investigation. The use of palm kernel shells, a waste product, not only offers an economical and sustainable solution but also reduces environmental pollution by repurposing waste materials for water treatment.

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