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Bio-Sorbent Optimisation Through Chemical Activation in The Processing of Waste Water in The Oil and Gas Sector

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ABSTRACT - The oil and gas industry is renowned for producing water that poses a significant threat to the environment, making its management a major concern. Therefore, the purpose of this research is to evaluate the efficiency of different biosorbents in purifying treated wastewater. The bio-sorbents used are derived from cashew shells (KMR), jenitri fruit (JNT), and walnuts (WN). This leads to the introduction of a new method, which combines different chemical activations with various bio-sorbents, alongside characterization and filtration performance evaluations. This results in high-performance bio-sorbents for produced water treatment. The methods adopted include adsorbent preparation, characterization (bulk density, FTIR, SEM), and filtration testing for turbidity and TDS parameters. The results show that chemical activation significantly enhances adsorbent performance, with candlenut shell + zinc chloride (KMR+ZC) and jenitri + potassium hydroxide (JNT+K) exhibiting the best adsorption capacity. Bulk density analysis shows a decrease in value after chemical activation, indicating increased porosity. Meanwhile, FTIR characterization confirms changes in functional groups after activation, with SEM showing structural modifications that improve the adsorption surface area. Filtration tests show that the modified adsorbents have better capacity in reducing turbidity and TDS in treated water..

Keywords: zinc chloride, potassium hydroxide, adsorption, filtration, bio-sorbent.

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INTRODUCTION

The oil and gas industry is the main constituent of the world economy (Sadikov, Radjibaev & Nizamov 2023), with the exploration and production activities responsible for the provision of energy, which has a significant impact on the environment (Haruna et al. 2023; Afdhol et al. 2019). However, the management of water produced during the extraction process along with oil and gas is a major issue (Makmur 2013), because it often contained harmful substances such as salts, heavy metals, and organic compounds (Klemz et al. 2021; Afdhol et al. 2020). The harmful substances contaminated soil and water sources, if not managed properly. This led to the need to understand the nature of produced water, including the effects on the environment and public health. Increased awareness of environmental issues, prompted effective management, and reduced the adverse impacts on the ecosystem. Additionally, strict regulations have been applied to produced water management in many countries (Effendi & Varayesi 2022; Aeruginosa 2016).

Based on the description above, bio-sorbents derived from agricultural and organic wastes have evolved as promising low-cost materials for water treatment purposes, specifically for adsorbing pollutants from wastewater streams (Karić et al. 2022). Several research had explored natural materials including coconut shells, sawdust, and fruit peels due to its high surface area and availability of functional groups that facilitated adsorption (Foo & Hameed 2012).

Considering the numerous research on chemically activated bio-sorbents, most existing analyses focused on a specific type of bio-sorbent or chemical activation agent. For example, Afdhol et al. (2025) examined bio-sorbents, particularly chemical activation agents. Ding et al. (2023) focused on optimized biochar activated with potassium hydroxide, using a specific type of biomass. In this context, comparative investigations on different types of natural materials and multiple activation strategies are limited, specifically in respect to wastewater treatment associated with the oil and gas sector.

This research addressed the diverse gaps by exploring the combined impact of the two chemical activation agents used, namely potassium hydroxide, and zinc chloride on three different bio-sorbent types including hazelnut shell (KMR), jenitri fruit (JNT) and walnut (WN). The uniqueness lies in its integrative method that centered on testing turbidity and TDS as well as performing detailed characterization through bulk density analysis, FTIR and SEM. This was aimed to understand the structural and functional changes resulting from chemical activation.

In line with the perspective above, new technology for treating and using generated water could be developed with the help of this analysis. Additionally, these technologies led to improved operational efficiency, and reduced expenses. The public may play a more active role in monitoring the effects of the oil and gas sector with a better understanding of the concerns. The main objective of this research is identifying which chemical effects absolutely retained adsorbates during filtration.

METHODOLOGY

The experimental procedure carried out started with adsorbent preparation, followed by characterization and filtration tests.

Adsorbent preparation

The initially obtained adsorbent was crushed to an average size of 0.5 cm. The crushed particles were sieved to obtain a uniform size using a multistage sieve with mesh 8, and 10. The final size was realized from mesh 16 as shown in Fig. 1. Furthermore, the uniform adsorbent was baked using Drying Oven DX302 for 60 minutes to remove the remaining water content. The product was activated with 30% KOH and 25% $ZnCl_2$ (Afdhol et al. 2025; Rosmayati 2015; Kane, Mishra & Dutta 2016), for approximately 60 minutes (Rosmayati 2015), and then washed until the pH returned to neutral. The produced water must not mix again with the content of chemical compounds used during the filtration test. Once the pH of the adsorbent had been confirmed to be neutral, it is dried again using an oven (Afdhol et al. 2023).

Adsorbent characterization

Bulk density

The bulk density test was conducted using the MM-0101 Tapped Density Meter. This variable refers to an essential measure of the mass of a material per unit volume. Bulk density takes into account the entire volume of the substance, including the empty spaces or gaps between particles. Furthermore, particle form, size, and distribution, including the porosity of the material, packing and compaction methods adopted, as well as the moisture content had an effect on the results of the test. Bulk density testing is a useful method in various industries due to the diverse characteristics (Altino, Lourenço & Ataíde, 2021 and Bernhart & Fasina 2009), and its values calculated using the following equation (Campiñez et al. 2016).

$$\rho_{bulk} = \frac{m}{V_{bulk}} \quad (1)$$

FTIR

FTIR characterization was performing using the Prestige-21 IR instrument manufactured by Shimadzu. Meanwhile, the interactions between materials and infrared light resulted in the adoption of an instrumental analysis method of fourier transform infrared spectroscopy (FTIR), used to identify chemical structures and functional groups. This characterization method played a crucial role in materials analysis and was widely used in various fields of science.(Siddique 2024)(M. K. Afdhol et al. 2023)

SEM

The SEM test was performed using a JSM-6460LA Scanning Electron Microscopy. This method was used to obtain surface images of material samples at the nanometer scale using a high-energy electron beam. SEM is among the most important tools in the fields of material science and engineering, as it provided information at the micro and nano scales which cannot be obtained using conventional microscopy methods (Schinazi et al. 2022).

Figure 1. Crushing adsorbent particles candlenut shell (KMR) after sieving

Filtration Test

Turbidity

Produced water was realized in conjunction with the oil and gas extraction process from wells, containing a variety of dissolved and suspended substances (Scanlon et al. 2020). The turbidity level represented the concentration of suspended particles that negatively impacted the environment and operational efficiency (Gogoi et al. 2020). Meanwhile, turbidity testing was performed using a measuring instrument called a turbidimeter. This instrument measures how far light is absorbed or scattered by particles in water sample. The unit is Nephelometric Turbidity Unit (NTU) which shows the intensity of water turbidity due to the presence of suspended particles (Faisal et al. 2016). The greater the measured NTU, the more turbid water condition was categorized, as an indicator of high concentration of suspended particles, minerals, microorganisms, or decomposed organic matter. Produced water with high turbidity causes problems such as pipe blockages, decreased efficiency of injection systems, and damage to production equipment (Chikwe & Ogwumike 2018). This test used 60ºC as the temperature of produced water, that flows into a filter funnel filled with adsorbent. Furthermore, water passing through the filtration funnel was measured for turbidity every five seconds. Equation 2 was used to calculate turbidity as follows (Faisal et al. 2016):

$$Turbidity\ (NTU) = \frac{I_0}{I} \times k \quad (2)$$

TDS

Total Dissolved Solids (TDS) refers to the amount of minerals, salts, metals, and organic compounds present in water. This was determined by measuring the electrical conductivity of the solution, directly related to the amount of ions contained, or by evaporation to determine the extent of solid dissolved.(Steininger et al. 2023). In the oil and gas industry, produced water was characterized by extreme high TDS concentrations due to its contact with rock formations during production. Some of the problems associated with high TDS concentrations were corrosion of production equipment, deposition in pipes, and pollution due to the discharge of untreated produced water. (Daniels & Feasey 2020). TDS measurement was performed with a portable meter, and equation 3 used to calculate the actual value from electrical conductivity (Mohit & Suprita 2022):

$$TDS\left(\frac{mg}{L}\right) = k \times EC\left(\frac{\mu S}{cm}\right) \quad (3)$$

RESULT AND DISCUSSION

Bulk density characterization

Figure 2, showed that the bulk density range of adsorbent produced from different natural materials varied significantly. Adsorbent KMR (candlenut shell) had the highest value of 0.76 g/cm³ which implied the material was relatively compact. This result was supported by Buhani et al. (2022) who stated that candlenut shell had a compact cellular structure with high content of fibrous lignocellulosic materials, resulting in the high density. However, when modified with $ZnCl_2$, the bulk density of KMR+ZC reduced to 0.73 g/cm³, implying that chemical activation changed the internal structure of the material.

WN (walnut) adsorbent with bulk density of 0.71 g/cm³ occupied the middle part of the graph. This was in line with the research by Foo and Hameed, (2012), that shell-based materials had bulk density characteristics due to the microstructure. Meanwhile, JNT (jenitri) adsorbent showed decreased bulk density of 0.68 g/cm³, associated with the changed internal structure of the material. Further modification with KOH, reduced the bulk density of JNT+K to 0.60 g/cm³. This significant decrease was in line with the research by Demiral et al. (2011) concerning the effects of chemical activation on the structure of the adsorbent.

Differences in bulk density had significant implications in adsorption applications. According to Kalaba et al. (2022), bulk density strongly correlated with adsorption capacity, specific surface area, and efficiency of the material to capture contaminants. This was consistently reduced by chemical modification with $ZnCl_2$ and KOH, which enhanced porosity and accessibility of active sites on the adsorbent's surface. Based on this context, chemical activation process was transformational on physical structure, and played an improvement role on the adsorptive capacity of materials such as candlenut shell, walnut, and jenitri.

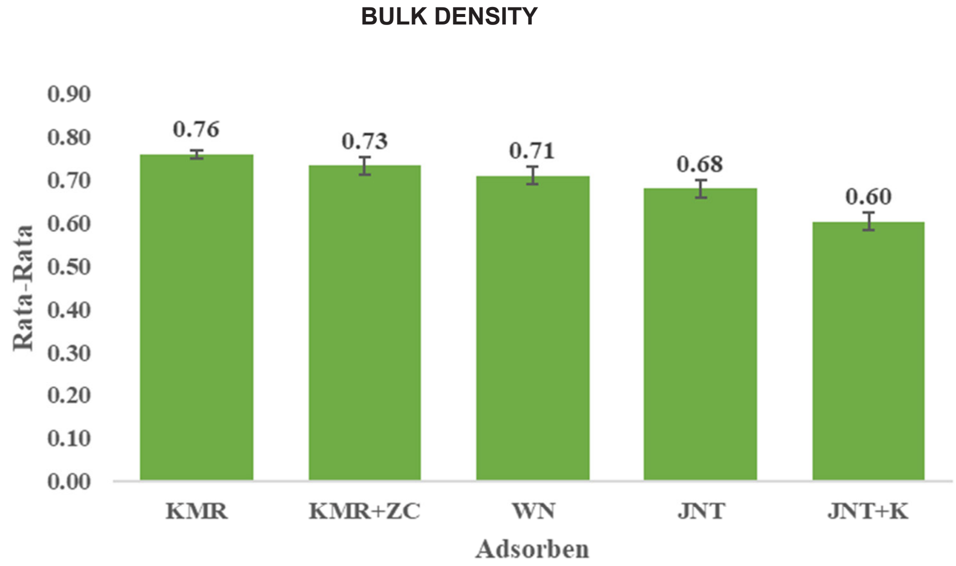

Figure 2. Bulk density test results of adsorbent

FTIR

Characterization of candlenut shell using FTIR

The results of the FTIR characterization in Fig. 3, showed a big difference between the spectra of candlenut and the shell with zinc chloride ($ZnCl_2$). The highest transmission peak in the candlenut shell spectrum was relatively 104-105%, while the spectrum with $ZnCl_2$ had a maximum intensity of approximately 87-90%. This difference shows chemical changes caused by the addition of zinc chloride catalyst. Munir & Swasono (2012), examined how the addition of metal catalysts affected the molecular structure of candlenut shell by interacting with existing functional groups.

The main peaks at different wavelengths offered a better understanding of the entire analysis. After the addition of $ZnCl_2$, the strength of the peaks recorded within 2850–2920 cm-1, due to the C-H stretching vibrations of methylene and methyl groups, was altered. However, a peak of relatively 1700 cm-1, representing the C=O bond of the ester carbonyl group, was also altered. This was consistent with the research by Teixeira et al. (2018), on candlenut shell. The result showed that changes in FTIR spectra led to the formation of metal complexes, alterations in molecular bonds, or other chemical changes. The 1000-1300 cm-1 and 3300-3500 cm-1 regions showed C-O and O-H bonds, respectively.

Considering the spectra analysis and evidence from previous research, candlenut shell was subjected to significant structural changes due to the addition of zinc chloride. The result had an impact on physicochemical characteristics such as reactivity, stability, and potential applications.

Characterization of walnut using FTIR

In Figure 3, fourier transform infrared FTIR of walnut adsorbent, showed characteristic absorptions. These showed the presence of chemical functional groups on the surface of the material.

Based on the analysis, the absorption peaks within 3113-3600 cm^{-1} were equivalent to the transmission interval of approximately 87-92%. This showed that the -OH bound was quite significant in the structure of cellulose and lignin. Moreover, a sharp peak with 87% transmission at 2128 cm-1 was attributed to C≡C bonds. The most significant peak was observed at 2400 cm-1 with a minimum transmission value of 81%. It implied that the absorption present in the -COOH bond remained unchanged.

The results of the FTIR analysis showed the presence of a fingerprint within 1800-400 cm^{-1}. This region was obtained by deriving increased %T (decreased absorption) while reaching absorption peaks of 1726 cm-1, 1590 cm-1 and 1033 cm-1 with transmission exceeding 80-85% in the C=0, C=C, and C-O bonds. The stronger transmission (>85%) less than 1000 cm-1 represented weak absorption by functional groups within that range.

The research by Ivanchenko et al., (2025), on the use of walnut shells as adsorbents, reported similar transmittance patterns. Additionally, strong absorption were detected at 3000-3600 cm^{-1} and

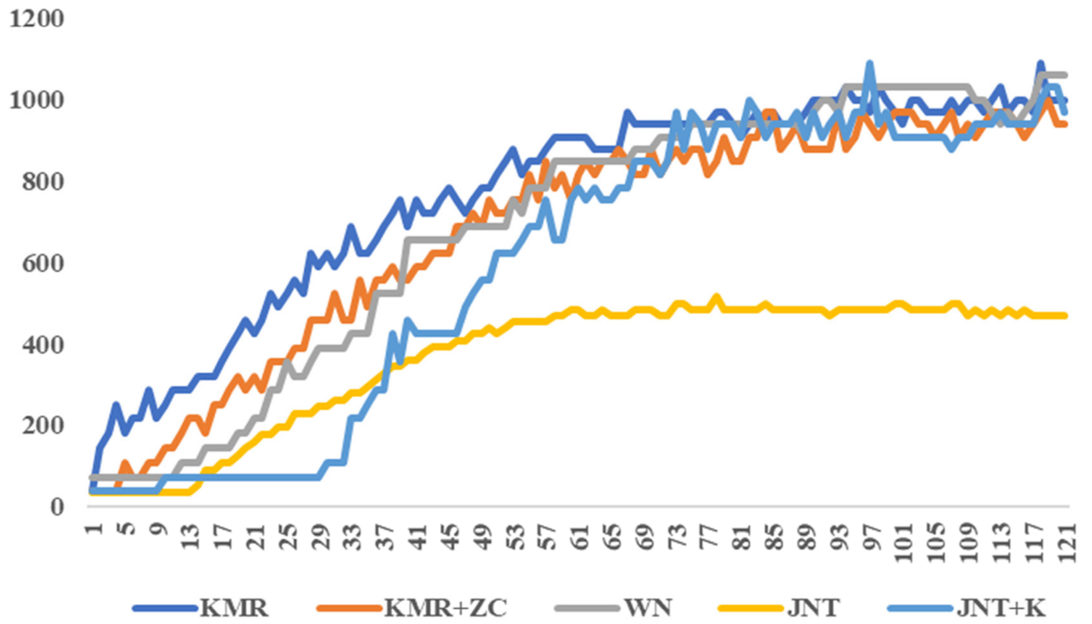

Figure 3. FTIR spectra of various bio-sorbent samples: KMR (blue), KMR+ZC (orange), WN (gray), JNT (yellow), and JNT+K (light blue)

1700-1730 cm⁻¹ which implied hydroxyl and carbonyl groups actively participated during the pollution adsorption process. Xu et al., (2022), reported that low %T values (high absorbers) found in the spectral region correlated with higher adsorption capacities. The research by Nguyen et al., (2014), stated that the comparison of %T values before and after adsorption process showed the presence of various functional groups during the mechanism. This was proven by alterations in the intensity of adsorptive transmittance, reflecting the interaction between the adsorbent and specific functional groups on the surface.

Characterization of jenitri using FTIR

The spectra of jenitri in Figure 3, showed that some peaks exhibited low transmittance (or high absorbance), such as at wave numbers 3363,08 cm^{-1}, 2891,42 cm^{-1}, and 1045,45 cm^{-1} with transmittance of approximately 88%, 81%, and 71%, respectively. These peaks with low transmittance showed strong absorption due to functional groups, which implied the presence of significant amounts of O-H, C-H, and C-O in the Jenitri adsorbent.

Regarding the spectra of jenitri+KOH, certain changes were observed in the transmittance pattern showing the modification of functional groups after KOH activation. The peak at 3654,30 cm^{-1} showed a transmittance of relatively 85%, lower than the O-H peak in pure Jenitri. This implied an increase in the number of hydroxyl groups due to KOH activation. Furthermore, the peak at 2891,42 cm^{-1} showed a transmittance of approximately 80%, similar to pure jenitri. This suggested that the C-H groups were not significantly affected by KOH activation.

Significant differences were observed within the 1600-1800 cm^{-1} region, where the carbonyl peak in jenitri (1734,06 cm^{-1}) had a transmittance of relatively 78%. Meanwhile, in jenitri+KOH, the peak shifted to 1675,23 cm^{-1} with a transmittance of approximately 83% found in C=O bonds. This change showed the reaction of carbonyl groups with KOH, caused the reduction in absorption intensity. In the fingerprint region, the peak at 1038,71 cm^{-1} in jenitri+KOH exhibited a transmittance of approximately 75%, which showed an increase in the number of C-O groups after KOH activation.

Ibrahim et al., (2023), reported that alkali activation such as KOH caused an increase in absorbance within the 3200-3600 cm^{-1} region (%T decrease), representing an increase in hydroxyl groups. This was consistent with the results observed in the spectra of jenitri+KOH. According to Ma, (2021), the change of transmittance pattern in the 1000-1800 cm^{-1} region after KOH activation showed the modification of aromatic structure and oxygen-containing functional groups, which improved adsorption capacity of the material.

Scanning electron microscopy (SEM)

Figure 4 (A) shows a candlenut shell characterized by layered surface with clear linear undulations and visible striations. The surface appeared relatively smooth with parallel elongated features suggesting the presence of well defined structured materials with potential efficient adsorption sites.

(B) is the candlenut shell + $ZnCl_2$ characteried by increased irregular and fragmented surfaces compared to the original candlenut shell. The zinc chloride treatment changed the surface topography into a more porous and rough granular landscape. These modifications increased the surface area of the material and the adsorptive capacity.

Figure 4 of (G)jenitri, (D)jenitri + KOH, and (E)walnut exhibited more complex and less orderly surface morphology. The material (C)jenitri had sponge like surface characterized by a congregation of cavities with multiple bulges and protrusions. The potassium hydroxide treatment increased surface roughness by producing more sharply defined edges and greater porosity.

In view of the description above, Figure 4 (E) walnut exhibited exaggerated roughness with folding surfaces superimposed on each other, giving it the relevant ability to capture adsorbent molecules. The hypothesized surfaces suggested that the material had significant complex topographies with multiple potential binding sites and larger surface area.

Differences in morphology was critical during adsorption because surface characteristics directly dictated the capacity, efficiency, and mechanisms of material adsorption. Earlier research by Foo & Hameed (2010) published in the Journal of Hazardous Materials reveals that the chemistry of biomaterials was proficiently modified to significantly enhance the adsorptive properties by generating additional active sites and increasing surface roughness.

The research by Babel & Kurniawan (2003), published in Journal of Hazardous Materials further supported the result by reporting that chemical modification of natural adsorbents improved the textural properties. This entailed enhancing the effectiveness in removing diverse contaminants from water solutions.

Filtration Test

Turbidity

In Figure 5, the results of the turbidity test showed that the five different adsorbents had varying capacities in terms of reducing water turbidity. Each adsorbent exhibited certain characteristics in adsorption process, evident in the growth curve

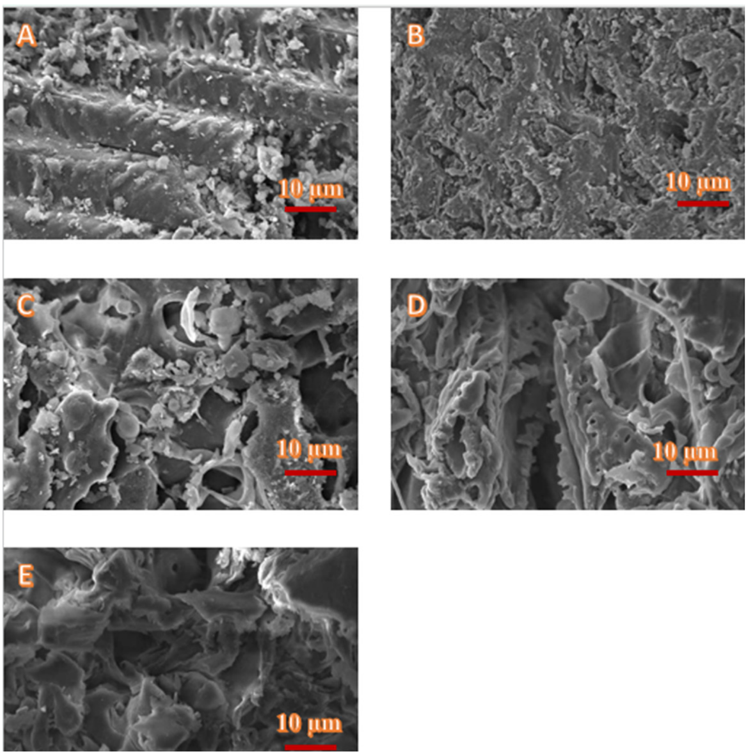

Figure 4. SEM characterization of (A)KMR, (B)KMR+ZC, (C)JNT, (D)JNT+K, (E)WN

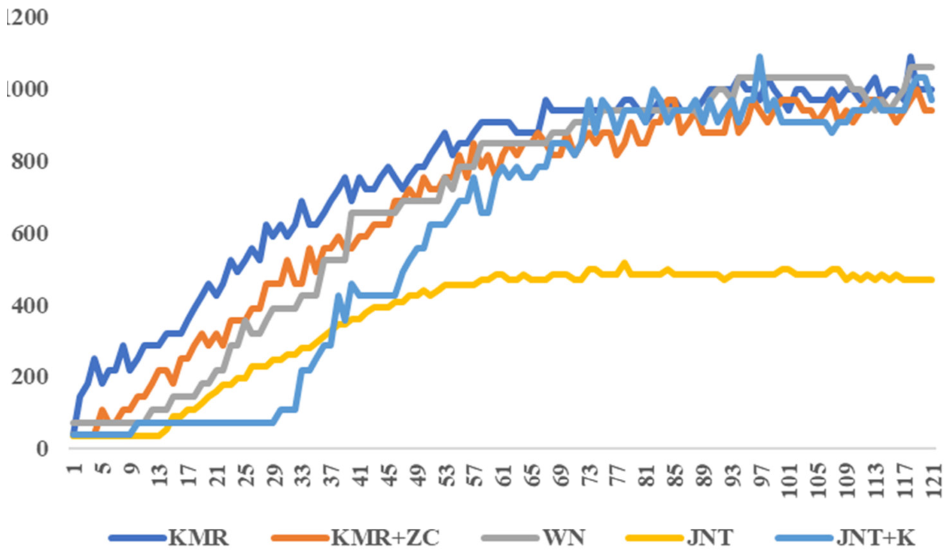

Figure 5. Turbidity test results of five bio-sorbents over time

of turbidity values over time. Candlenut shell adsorbent exhibited the most dynamic performance, characterized by rapid increase in turbidity and a maximum value of 1088 NTU obtained at the end of the test. This showed a low adsorption capacity, which implied the inability of the adsorbent to bind the turbidity-causing particles properly. However, the candlenut shell with $ZnCl_2$ which had a value of 900 NTU showed better performance than pure candlenut. The resulting turbidity curve showed a more gentle slope, representing better adsorption ability. Theoretically, changes including metals such as zinc chloride increased the surface area and reactivity of the adsorbent. The research by Ningsih, (2018) supported the phenomenon that the addition of more $ZnCl_2$ opened the cavity of activated carbon, increasing its adsorption ability. Based on the turbidity curve showing intermediate adsorption properties, the walnut adsorbent with a value of 1060 NTU reached the highest turbidity value at the end. Jeniter showed consistent but limited adsorption ability, with a relatively flat and stable curve at 500 NTU. However, the KOH-modified jenitri adsorbent exhibited the most interesting performance. Adsorption characteristics were significantly altered due to this alkali modification, and the curve was more controllable than the former. According to Ding et al. 2023), alkaline treatment had the ability to change the shape of the adsorbent surface. This occurred by increasing the active fungal groups, which allows better binding of contaminant particles. The significant difference between the pure and modified adsorbents suggested that chemical interventions such as the addition of metals ($ZnCl_2$) or strong bases (KOH) significantly improved adsorption ability.

TDS

Figure 6 shows an increase in TDS over time, with different patterns for each material. However, when compared with other materials, pure candlenut, jenitri+KOH, and candlenut shell+$ZnCl_2$ were the best in adsorption, with a gradual increase in TDS. This showed that chemical modification played an crucial role in improving adsorption capacity. The addition of zinc chloride to candlenut shell and KOH to jenitri increased the capacity of the materials to reduce TDS significantly at a value of 180 mg/L. The result was in line with previous research by Rafatullah et al. (2010), that chemical treatment increase the surface area and active sites on natural adsorbents. In addition, Foo & Hameed (2010) stated that chemical alteration affected the surface structure and properties of the material, leading to increased adsorption efficiency. Jenitri and walnut showed faster increasing TDS with final results of 430 mg/L and 640 mg/L, respectively representing lower adsorption capacity. This was caused by poor surface structure and lack of active sites to capture solutes. The comparison between the original and modified materials showed that chemical pretreatment was essential in optimizing adsorbent performance. The surface characteristics of natural materials were changed by chemical modification, improved ion exchange and adsorption abilities (Crini 2006). This was consistent with the pattern shown in the graph,

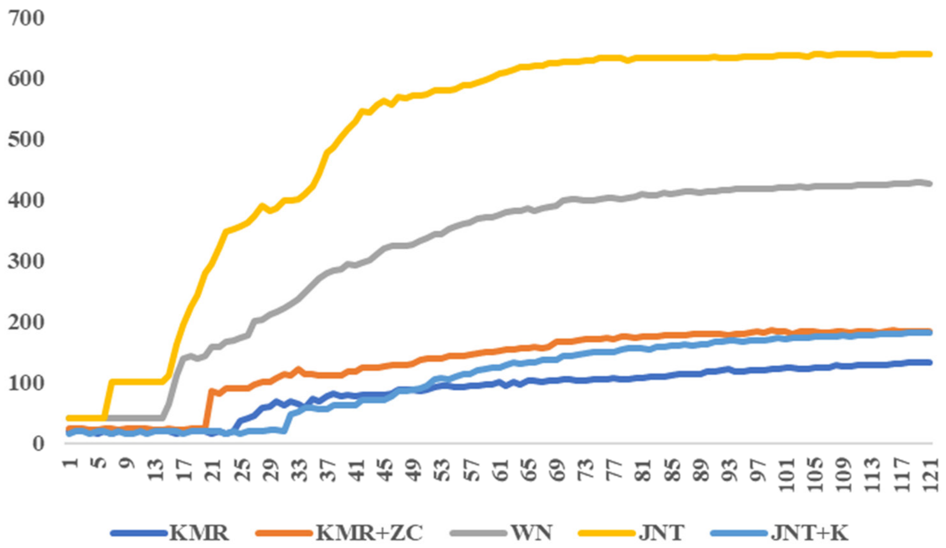

Figure 6. TDS test results of some adsorbents

where jenitri+KOH and candlenut shell+$ZnCl_2$ exhibited better performance than the starting materials. Meanwhile, the changes in chemical structure was accountable for the difference, allowing for effective interaction with the solute. The results showed that chemical modification with $ZnCl_2$ and KOH significantly improved adsorption ability. This implied natural materials had the ability to serve as effective adsorbents after proper chemical treatment, thereby creating opportunities for the development of sustainable water treatment solutions that are environmentally friendly.

Regarding the results of the TDS and turbidity tests performed on the adsorbents, JNT+K and KMR+ZC had good values, compared to data obtained from various sources in Table I. The modifications made in this research caused the characterization process to exceed the other adsorbents.

Table 1. Comparative analysis of adsorbent ability to reduce turbidity and TDS

Adsorbent	Turbidity Reduction (%)	TDS Reduction (%)	Notes	Ref
Graphene Oxide–Keratin–Chitosan Nanocomposite	86%	29%	Effective at pH 6, dosage 2 g/L, contact time 25 min	(Roy *et al.*, 2023)
Natural Coagulants (e.g. Moringa Oleífera)	23.07%	Not Specified	Comparable to aluminum sulfate	(Alcántara Llovera and Silva-Chuquipoma, 2023)
Bioflocculants (BFs)	86%	Not Specified	Effective with dosage 6-20 mg/L, enhanced with Al or Fe	(Ma, Zheng and Chi, 2008)
Phyto-Coagulants (e.g. White Popinac)	76%	Not Specified	Effective at dosage 50 mg/L, initial turbidity 319 NTU	(Al-Mamun and Basir, 2016)

Table 1. Comparative analysis of adsorbent ability to reduce turbidity and TDS (continued)

Adsorbent	Turbidity Reduction (%)	TDS Reduction (%)	Notes	Ref
JNT+K	High 95%	High 97%	Highly effective in adsorbing oil and reducing turbidity in produced water.	
KMR+ZC	High 94%	High 95%		

CONCLUSIONS

In conclusion, this research showed that the use of chemically modified natural adsorbent materials increased the effectiveness in reducing turbidity and TDS in produced water. Additionally, candlenut shell modified with $ZnCl_2$ and KOH-modified jenitri were shown to exhibit better adsorption capacity compared to the pure adsorbent. Chemical modification altered the adsorbent's physical structure and enhanced its reactivity and adsorption capacity, crucial for the management of generated water in the oil and gas sector. These results provided chances for sustainable water treatment solutions, adequate for the environment. Furthermore, the results also got rid of the need to import walnuts raising awareness of how the oil and gas sector affected the environment.

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GLOSSARY OF TERMS

Symbol	Definition	Unit
KMR	Candlenut Shell	
KMR+ZC	Candlenut shell activated with $ZnCl_2$	
WN	Walnut	
JNT	Jenitri	
JNT+K	Jenitri activated KOH	
$ZnCl_2$	Zinc Chloride	
KOH	Potassium Hydroxide	
ρ_{bulk}	Bulk Density	gr/cc
FTIR	Fourier Transform Infrared Spectroscopy	
SEM	Scanning Electron Microscopy	
TDS	Total Dissolved Solids	mg/L
NTU	Nephelometric Turbidity	

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