

Silica Nano-particle Polyethylene Glycol Nano-composite Hydrogel as Adsorbent for Bleaching Palm Oil

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Abstract: Bleaching vegetable oil is one of the ways of removing impurities and pigments in oil for industrial and domestic applications. It is an adsorption process where the particles clog on the adsorbent surface. This work looks at bleaching Palm Oil using Silica Nanoparticle (SiO₂ Nps) approximately 5 nm in size mixed with Polyethylene Glycol (PEG) to form SiO₂ Nps-PEG hydrogel nanocomposite. From the experiment, the diffraction peaks 102, 200, 101, 101, 212, 301, 110, 211, and 123 at Bragg's angle 15°, 17°, 21°, 23°, 26°, 27°, 36°, 39° and 43° from X-ray diffraction (XRD) analysis, shows that amorphous SiO₂ was formed and peak M specifies the presence of PEG in the SiO₂ Nps-PEG nanocomposite hydrogel. Scanning Electron Microscope (SEM) analysis showed a well dispersed SiO₂ in the SiO₂ Nps-PEG hydrogel nanocomposite compared to aggregated SiO₂ Nps without PEG. The SiO₂ Nps-PEG hydrogel effectively bleached raw palm oil indicated by an increase in fatty acids from 86.6% in the unbleached oil to 97.1% in the bleached oil; increase in the aldehydes from 0.67% in the unbleached oil to 0.9% in the bleached oil; decrease in alkanones from 2.79% in the unbleached oil to 0.94% in the bleached oil; decrease in other organics from 9.38% in the unbleached oil to 0.48% in the bleached oil and finally alkene increase from 0.21% to 0.34% as obtained from the Gas Chromatography/Mass Spectrometry (GCMS) analysis. This shows that the SiO₂ Nps-PEG nanocomposite hydrogel is an effective agent in bleaching palm oil.

Keywords: Silica nanoparticles; polyethylene glycol; hydrogel; palm oil; bleaching

1. Introduction

From ancient times, vegetable oil has been seen to hold great value as it has its application from cooking to cosmetics and other products in chemical industries. Vegetable oil production over the years have not just increased, but the demand for high quality vegetable oil have become essential as vegetable oil with fine appearance and less contaminants is required for better health and excellent chemical processes. One of such vegetable oil is palm oil which is readily available in West Africa especially the southern part of Nigeria. Palm oil is extracted through chemical, mechanical or traditional methods from palm bunch fruits. The extracted oil have been used in many food applications such as cooking oil, frying, vegetable ghee, margarines etc. and in cosmetics productions and perfumes.^[1] Research has also shown that palm oil is beneficial health wise when consumed.^[2,3] The application of palm oil depends on the quantity of contaminants in it. Contaminant in oil originates from natural sources, while others are as a result of the processing conditions in the oil refining process. For food oil, contaminants presents includes Phospholipids, free fatty acids, pigments, traces of metals, oxidation products, persistent organic pollutants (POPs), volatiles and many more which depends on processing conditions.^[4] To attain high grade oil, bleaching of the oil is key, as this process reduces the colour of the oil as well as removing or converting unwanted by-products to harmless forms. These guarantees the non-interference of the

unwanted compounds with the processing of the oil as required for human consumption and industrial applications.^[5] The removal of contaminants is made possible by their adherence to the surfaces of suitable adsorbents; bleaching therefore takes place when an adsorption mechanism is present. Adsorption is a process where ions, molecules or atoms from a liquid, gas or solids dissolved in solution adhere to a surface. In bleaching food oil, activated bleaching earth or active carbon are the frequently used adsorbents. Bleaching earth is known to have a good absorbent capacity which enables it to removes impurities and contaminants, but the residual oil recovery is uneconomical as the unused bleached earth containing fats are deposited as waste causing environmental problems.^[6] Another key adsorbent often used in bleaching food oil is the activated carbon. It is an absorptive substance with large surface area in excess of 3000 m² used widely in adsorption.^[7] Activated carbon has its drawbacks such as the inability to bind very well with diols, alcohols, strong bases, strong acids, metals, inorganic substances such as sodium, lead, lithium, boric, fluoric acids etc. They are useful for removing pigments such as xanthophyll, chlorophylls, and carotene. Other techniques applied for bleaching palm oil are; activated clay or acid activated clay methods but these methods are expensive, unreliable and less environmental friendly.^[8] These drawbacks in the effectiveness of these adsorbents, has made researchers to look for alternative ways of to improve on adsorption efficiency of the adsorbents. The application of nanoparticles and its



Fig. 1. (a) Sol-gel Formation in SiO_2 and (b) SiO_2 Nps synthesized



Fig. 2. SiO_2 -PEG Nanocomposite Hydrogel

nanocomposite are possible alternatives in the adsorption of impurities and pigments in food oil. Nanoparticles are materials with sizes within 1 to 100 nanometers. One of such materials is silica nanoparticles (SiO_2 Nps) known for its unique properties such as high pore volume, large surface area, easy functionalization, tunable pore size and uniformity^[9,10] and as a good bleaching agent.^[11,12] These unique properties of SiO_2 Nps can be improved by combining it with polymeric materials and one of such polymeric material with unique properties is polyethylene glycol (PEG). In this research work, SiO_2 Nps is combined with PEG to form a nanocomposite hydrogel (SiO_2 Nps-PEG) to bleach palm oil. Hydrogels are known as a network consisting of non-fluid polymer whose volume is expanded by water.^[13] And PEG is a non-toxic, biocompatible, biodegradable synthetic hydrophilic polymer.^[14] The hydroxyl group present within the SiO_2 Nps-PEG composite structure due to PEG will create the ideal environment to attract polar impurities from the oil to the nanocomposite surface and taking advantage of the large surface area of SiO_2 Nps. Contaminants such as soaps, phospholipids and trace metals will be drawn into the pores of the nanocomposite structure. Thus the synergy from high surface area from SiO_2 Nps and high absorptive volume from PEG will give SiO_2 Nps-PEG nanocomposite hydrogel superior adsorption capacity and eliminate the drawbacks in other methods used for bleaching oil.

2. Experimental Section

The equipment and materials used for this research are; beaker, stirrer, electric heater, mass balance, measuring cylinder, burette, funnel, centrifuge and pH meter, XRD (x-ray diffractometer), SEM (scanning electron microscope), GCMS (gas chromatography/mass spectrometer). Sodium Silicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$), ammonia (NH_3 (33% extra pure)), distilled Water (H_2O), ethanol (analytical grade), polyethylene glycol (PEG (4000 Mw)), Unbleached Palm Oil.

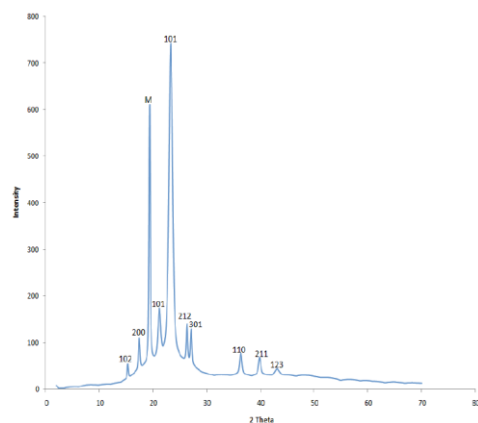


Fig. 3. XRD analysis of SiO_2 -PEG Nanocomposite Hydrogel

2.1. Preparation of Silica nanoparticles

Sol-gel method was applied in synthesizing SiO_2 Nps. The steps follow similar procedures used by Zulfiqar and co-researchers.^[15] A mixture of 30 ml Ammonia and 30 ml ethanol was prepared. 28.42 gram sodium trisilicate was dissolved in 245 ml of water (distilled) to produce sodium silicate solution. This sol-gel method was carried out in an alkaline medium and the sodium silicate solution had a pH range of 11-12. 2 ml sodium silicate solution drop wise was added into the ammonia/ethanol solution and allowed to age for one hour. The finished product was centrifuged at 4000 rpm for 30 minutes and then washed with water. The washed product was dried by heating to obtain SiO_2 Nps.

2.2. Preparation of SiO_2 Nps-PEG Nanocomposite Hydrogel

The SiO_2 Nps produced was mixed with 30 ml of ethanol and heated to dryness; this was done to remove the excess Ammonia. In making the nanocomposite melts, 0.13 gram of Silica nanoparticles was added to 30 ml of ethanol and then, 1.3 gram of PEG was introduced; the mixture was heated at 60°C and mixed thoroughly. On heating, distilled water (5 ml) was added, and the mixture kept under ambient condition to dry. The resulting SiO_2 -PEG nanocomposite hydrogel was pulverized and characterized using the XRD and SEM analysis.

2.3. Procedure for Bleaching the Palm Oil

20 ml of raw unbleached palm oil was heated at 60°C for 5 minutes and allowed to cool; this was done to remove all volatile impurities, 3.2 gram of SiO_2 Nps-PEG nanocomposite hydrogel was introduced into the oil and the mixture was covered for 6 hours. The oil was filtered and analyzed using the GCMS analysis.

3. Results and Discussions

The Fig. 1a shows the Sol-gel technique used in synthesizing SiO_2 Nps and Fig. 1b shows the SiO_2 Nps synthesized through the Sol-gel method. Fig. 2 shows the SiO_2 -PEG nanocomposite hydrogel synthesized. Comparing Fig. 2 and Fig. 1b showed that Fig. 2 had larger particle components compared to the finely pulverized SiO_2 Nps; the reason for this difference is because of the PEG which helps in the formation of the gel. Fig. 3 shows XRD analysis of the

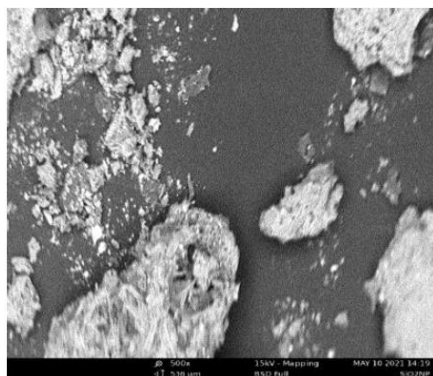


Fig. 4. SEM image for synthesized SiO₂ Nps

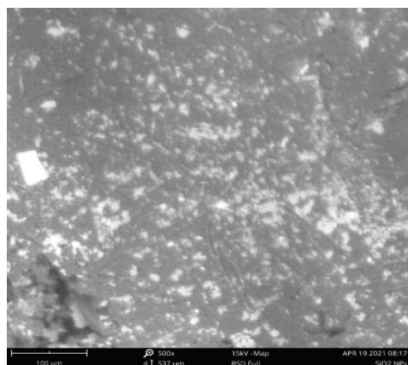


Fig. 5. SEM image for SiO₂-PEG Nanocomposite Hydrogel



Fig. 6. Bleaching of palm oil with SiO₂-PEG Nanocomposite Hydrogel

synthesized SiO₂-PEG nanocomposite hydrogel. The diffraction peaks in Fig. 4 indicated by miller indices 102, 200, 101, 101, 212, 301 and 110 at Bragg's angle 15°, 17°, 21°, 23°, 26° and 27°, 36° respectively, confirms the formation of Amorphous SiO₂ Nps based on The Joint Committee on Powder Diffraction Standards with Card number 39-1425 (JCPDS 39-1425). Also XRD peaks 211 and 123 at angle 39° and 43° respectively shows similar amorphous SiO₂ Nps (JCPDS 41-1413). The average amorphous SiO₂ Nps size was calculated to be 4.5317 nm applying the Bragg's equation shown in equation 1.

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

This result was corroborated with the SEM scan shown in Fig. 4 and Fig. 5. Fig. 4 shows a dense structure / aggregated structure of SiO₂ Nps. This might be due to the absence of a stabilizing agent during the preparation of the SiO₂ Nps or the length of time it took for the SEM analysis of the nanoparticles.

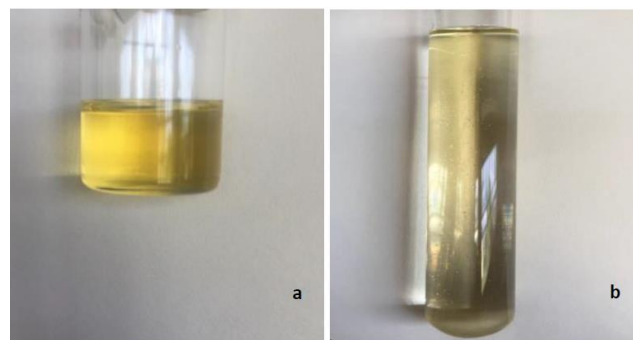


Fig. 7. (a) Palm oil before bleaching (b) palm oil after Bleaching

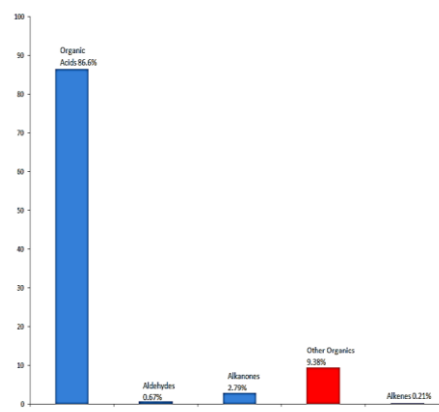


Fig. 8. GCMS Analysis of unbleached palm oil

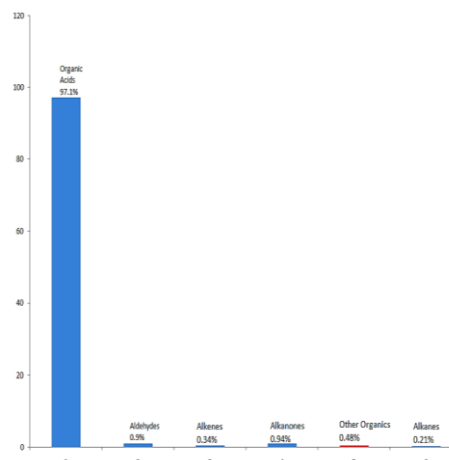


Fig. 9. GCMS analysis of bleached palm oil

Fig. 5 shows the surface morphology of SiO₂-PEG nanocomposites hydrogel. The Figure shows a dispersed SiO₂ Nps in PEG. This is because PEG acts as a stabilizing agent preventing the nanoparticles from aggregating.

Fig. 6 shows the bleaching process for the raw palm oil containing the SiO₂-PEG composite hydrogel. The SiO₂-PEG hydrogel/palm oil mixture was kept for six hours under ambient condition.

Fig. 7 (a) and (b) shows the unbleached and bleached oil using the SiO₂-PEG nanocomposite hydrogel.

Comparing Fig. 7 (a) and (b) it was observed that the bleached oil looks slightly colorless compared to the unbleached oil. This might be

an indication of the removal of impurities. This result was confirmed from the analysis obtained using GCMS on the unbleached and bleached oil. Fig. 8 shows the tabulated components for the unbleached oil obtained from using the GCMS analysis, and Fig. 9 shows the tabulated component for the bleached oil. In comparing the two figures, there is an increase in fatty acids from 86.6% in the unbleached oil to 97.1% in the bleached oil; increase in the aldehydes from 0.67% in the unbleached to 0.9% in the bleached oil; decrease in alkanones from 2.79% in the unbleached oil to 0.94% in the bleached oil; decrease in other organics from 9.38% in the unbleached to 0.48% in the bleached oil; Alkene increase from 0.21% to 0.34% and finally alkane presence in the bleached oil of 0.21%. This confirms that the SiO₂-PEG nanocomposite hydrogel effectively removed impurities from the unbleached palm oil.

4. Conclusions

Conclusively, SiO₂ Nps was successfully synthesized using Sol-gel technique and also SiO₂-PEG hydrogel was formed. The XRD result confirms the formation of the SiO₂ Nps and its composite hydrogel. The SiO₂-PEG hydrogel was successfully used to bleach palm oil and the result showed an increase in fatty acids from 86.6% in the unbleached oil to 97.1% in the bleached oil; increase in the Aldehydes from 0.67% in the unbleached oil to 0.9% in the bleached oil; decrease in alkanones from 2.79% in the unbleached oil to 0.94% in the bleached oil; decrease in other organics from 9.38% in the unbleached oil to 0.48% in the bleached oil and finally Alkene increase from 0.21% to 0.34%, which is an indication of an effectively bleached oil.

Conflicts of Interest

The authors declare no conflict of interest.

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