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Regeneration Potential of Eggshell Adsorbent on Spent Vegetable Oil: A Mean to Achieving Global Sustainability

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Abstract: ***The regeneration potential of activated eggshell adsorbent (AESA) on spent vegetable oils (SVOs) is a promising approach to realizing global sustainability. Eggshell, which are typically discarded, was converted into an effective adsorbent in removing impurities from spent vegetable oil. This involved the cleaning and drying of eggshell followed by pulverization and chemical treatment for better modification, and improved adsorption capacity. Batch adsorption method was adopted at dosage range of 4 to 20 g at 4 g interval, and optimum time and temperature of 80 min and 50 ^{o}C respectively. Oil and adsorbents were mixed using magnetic stirrer and heated for 80 minutes at 50 ^{o}C. The adsorbent was separated from the oil by filtration of hot suspension on Buchner's funnel with filter paper in a vacuum at (10-15 mm Hg) for 2 hours. The cool filtrates were stored in sealed bottles for further analysis. The results revealed that at the optimum dosage of 16 g, the percentage recovery of free fatty acids (FFA) and peroxide (PO) was 91.63 and 87.11 % respectively. The adsorbent was able to turn the dark brown colour of SVOs to golden brown, and the objectionable odour becomes less objectionable. Overall, the adsorbent was able to regenerate the SVOs close to the original status, thereby providing an innovative solution that contributes to global sustainability, by reducing waste, conserving resources, and promoting circular economy.***

Keywords: Adsorption, Adsorbent, Modification, Regeneration, Sustainability,

I. INTRODUCTION

Vegetable oil is triglycerides extracted from the seeds of some plants. Triglycerides are esters generally derived from the reactions between glycerol and fatty acids. At a high temperature, fresh frying oils undergo some changes leading to the formation of some products which results in oil quality reduction (Onawumi *et al.*, 2017). During storage, heating and frying processes, the qualities of oil are affected as a result of oxidation which further result in degradation of the quality via polymerization and isomerization. According to Onawumi *et al.* (2017) reported the products are formed to include, oxides, higher oxides, non-functional organic compounds containing oxygen, free fatty acids, chain and cyclic hydrocarbons, and free radicals. Sangoremi *et al.* (2025) also noted that the continuous reuse of spent oil poses a serious health risk. When oil undergoes oxidative reaction, it causes destruction of fat-soluble vitamins, hence, losing the fat's nutritional components (Sangoremi *et al.*, 2025)

Spent vegetable oils (SVOs) are widely utilized in food handling both industrially and at home, and fried food items are quite possibly one of its biggest applications. Deep-vegetable oil frying is a method of drenching food in hot oil with a contact between oil, air and food at high temperature of 150 ^{o}C to 190 ^{o}C (Biag *et al.*, 2022; Zhang *et al.*, 2022). Fried food varieties taste really attractive, have a desirable texture and fresh surface, which make fried food sources exceptionally famous among customers (Szabo *et al.*, 2022). The egg shell (EGS) is the outer covering of egg, which is usually thin, fragile, and bristle. It is primarily composed of calcium carbonate, along with small amounts of protein and other minerals (Ahmed *et al.*, 2021). The colour of an EGS varies, depending on the species of bird that laid it, but it is commonly white or off-white (Ahmed *et al.*, 2021). The EGS serves as a protective barrier that encases the egg's content, providing support and shielding the delicate internal structure. Despite its delicate appearance, the EGS is actually quite strong and capable of withstanding the weight of an incubating bird sitting on it (Ahmed *et al.*, 2019a). However, it is still susceptible to cracking or breaking under pressure (Ahmed *et al.*, 2019a, Ahmed *et al.*, 2021). The surface of an EGS is covered with tiny pores that allow for the exchange of gases. These pores enable the developing embryo inside the egg to breathe by allowing oxygen to enter and carbon dioxide exits. In terms of texture, the outer surface of an EGS can feel smooth, but it may also have a slight roughness or a grainy texture (Ahmed *et al.*, 2019a). The texture can vary depending on the species of bird and individuals. Eggshells (EGS) have been utilized for various purposes beyond their role in protecting the developing embryos.

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They are sometimes crushed into a fine powder and used as a calcium supplement or fertilizer. EGSs have also been employed in art and craft, as well as in traditional remedies and folk practices (Ahmed *et al*., 2021). Finally, EGSs have been employed in various environmental remediation technologies such as removal of heavy metals, adsorption of radioactive meals, adsorption of total nitrogen, fluoride and phosphorus from wastewater (Lu *et al*., 2017; Ahmed *et al*., 2021; Onawumi *et al*., 2021). The present study focuses on the remediating potential of adsorbent derived from eggshell on spent vegetable oil.

II. MATERIALS AND METHODS

A. *Procurement and Preparation of Samples*

The egg shells were gathered from two eateries: Mega Kitchen Igbonna and Chicken Republic, Fakunle, Osogbo, Osun State, Nigeria. The samples were washed completely with water sourced from the research facility and flushed severally with distilled water to eliminate dirt. The samples were sun-dried for 24 hours and dried in an oven at 105 ^{o}C until a constant weight was achieved for 5 hrs and, were permitted to cool in desiccators. The dried samples were ground and sieved through 0.3 mm mesh size (Onawumi *et al*., 2017, 2021).

B. *Modification of agro-wastes*

The technique described by Bello *et al*. (2017) was modified by increasing the molarity of phosphoric acid (H_3PO_4) from 0.3 M to 0.5 M. An accurately weighed 14.0 g pulverized eggshell was placed into Erlenmeyer flask containing 250 cm^3 of 0.5 M phosphoric acid. The substance of the container was entirely blended and warmed on a hot plate until a thick paste was formed. The paste of eggshell was transferred into a crucible which was set in a furnace and warmed at 500 ^{o}C for 60 minutes. The samples were allowed to cool and afterward washed severally with distilled water to a pH of 7.80. Accordingly, the samples of pulverised eggshell were oven dried at 105 ^{o}C for 5 hours and the adsorbents were put an air tight container for additional analysis and usage (Bello *et al*., 2017).

C. *Preparation of used vegetable oil*

KING'S vegetable oil was acquired at a local market, Osogbo, Osun state, Nigeria. The manufacturing and expiry dates were ascertained. The oil was divided into two different portions and labeled. The first portion was the vegetable oil that was not used, the second portion was used in frying iced fish for about 10 times. The following parameters were analysed from all the portions: free fatty acid (FFA), peroxide value (PV), colour, odour.

D. *Batch Adsorption Studies*

The batch adsorption studies were investigated at varying conditions. The methods used by Onawumi *et al.* (2017), Ushedo *et al.* (2022) for the recovery of spent vegetable oils (SVOs) were employed. The oil samples were mixed with dried powdered eggshell adsorbent at ratio of 100 ml: 4 g (v/w). Oil and adsorbents were mixed using magnetic stirrer and heated for 80 minutes at 50 ^{o}C. The adsorbent was separated from the oil by filtration of hot suspension on Buchner's funnel with filter paper in a vacuum at (10-15 mm Hg) for 2 hours. The cool filtrate was stored in sealed bottle for further analysis. A number of experimental variables such as adsorbent dosage in the range of (4-20 g), contact time (20-100 minutes) and Temperature (30-70 ^{o}C) were studied on FFA, PO, colour and odour. The adsorbed amount of oil at equilibrium, q_e (mg/g) was determined out by the expression:

$$q_e = \frac{(Co - Ce)V}{W} \qquad 1$$

Where Co and Ce (mg/L) are the initial and equilibrium concentrations of the oil parameters under investigation, W (g), weight of adsorbent used and V is the volume of oil samples in liter.

The percentage impurities removal was determined thus:

$$\%R = \frac{(Co - Ce)100}{C_o} \qquad 2$$

E. *Effects of Adsorbent Dosage*

Different dosages of pulverized eggshell adsorbent (PESA), (4, 8, 12, 16, 20 g) were weighed and added to 100 ml of UFVO put into different Erlenmeyer flasks at optimum conditions. Oil and adsorbents were mixed using magnetic stirrer and heated for 80 minutes at 50 ^{o}C. The adsorbent was separated from the oil by filtration of hot suspension on Buchner's funnel with filter paper in a vacuum (10-15 mm Hg) for 2 hours. The cool filtrates were stored in sealed bottles for further analysis.

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III. DETERMINATION OF PHYSICOCHEMICAL PROPERTIES OF THE ADSORBENTS

A. Determination of Moisture Content

Moisture content (MC) Was obtained using standard methods ASTM D 2974 (2014). 2 g of the PESA samples was weighed into the crucible. This was oven dried at 105 ^{o}C to steady weight and kept in a desiccator. The percent MC was stated numerically as in (Boadu *et al*., 2018).

$$\%MC = \frac{C-D}{C-B} x100 \qquad 3$$

Where:
B = Weight of crucible (g)
C = Weight of crucible plus wet sample (g)
D = Weight of crucible plus dried sample

B. pH Determination

Exactly 3 g of the PESA sample was measured and drenched into 30 ml of boiled deionized water, and preserved for 24 hrs. The combination was mixed to guarantee proper dilution and separated. The pH of the subsequent filtrate was evaluated utilizing a computerized pH meter, Jenway 3520 (Boadu *et al*., 2018; Ebelegi *et al*., 2022).

C. Bulk Density

The standard strategy utilized in examining bulk density (BD) was from Ijaola *et al*. (2013) and Ebelegi *et al*. (2022). 5 g of the sample was put into a pre-weighed 5 ml measuring cylinder (w1). The cylinders were delicately tapped to take out air spaces within the samples in the cylinders to give a potential close pack (PBD). The volume occupied by the samples and the additional load in the cylinders were resolved utilizing analytical weighing balance and was recorded as (w_2). The BD was determined as:

$$BD = \frac{w_2 - w_1}{v} \qquad 4$$

Where:
BD = Bulk density (g/cm^3)
w_2 = weight of samples and cylinder (g)
w_1= weight of measuring cylinder (g)
v = Volume of the cylinder

D. Ash Content

Ash content (AC) entirely determined using method depicted by Ebelegi *et al*. (2022). 5 g of dried PESA samples was weighed into a crucible of a known weight and warmed in a muffle furnace for 6 hours at 600 ^{o}C. The crucible was permitted to cool in desiccators, and weighed until consistent weight was accomplished. The mass of the ashed carbon was noted. The weight of ashed carbon was calculated as percent weight of the original carbon sample.

$$\%TA = \frac{C-B}{D-B} x100 \qquad 5$$

Where:
TA = Total Ash
B = Weight of the crucible (g)
C = Weight of crucible + original sample (g)
D = Weight of crucible + ashed sample (g)

E. Volatile Matter

Volatile matter (VM) was calculated by method depicted by Ebelegi *et al*. (2022), accurately 1 g of samples the adsorbent was taken in a pre-dried crucible and covered with lid, then heated in a Gallenkamp muffle furnace controlled at 950 ^{o}C for 7 minutes. In the wake of heating, the plate was immediately covered, cooled in desiccators and weighed. The distinction in the first sample and residue is the volatile matter.

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$$\%Vm = \frac{C-D}{C-B} x100 \quad 6$$

Where:
VM = Volatile matter

B = Weight of the crucible (g)
C = Weight of crucible + sample (g)
D = Weight of crucible + residue (g)

$$\%VM = \frac{C-D}{C-B} x100 \quad 7$$

F. Particle Size

Particle sizes (PS) of the PESA samples was calculated. The samples were arranged utilizing electric blending machine after which a sifter examination was completed utilizing Controls Miland-Italy D402-01 Matr 84000 109 sieve shaker at rotation of 10-15 min with (75, 150, 300, 600, 750 µm) sieves (ASTM D-2862-97).

$$PS = \frac{w_1}{w_2} \quad 8$$

Where:
PS = Particle size
W_1 = Weight of the carbon after sieve
W_2 = Total weight of carbon

G. Surface Area

The surface area (SA) of the adsorbent was resolved utilizing the Sear's method, portrayed by Ebelegi *et a*l. (2022) where 0.5 g of each sample was painstakingly weighed into 250 ml conical flask containing 25 ml of 0.1M HCl at pH 3.50, after which 1 g of NaCl was added to raise the pH to 4, the blend was titrated against a standard solution of 0.1M NaOH until pH 9 was accomplished. The volume expected to build the pH from 4 to 9 was noted and utilized in processing the surface area utilizing Eq. 9.

Surface area (m^2/g) = $32V_{-}25$ 9

where, V is the volume of NaOH used to raise pH from 4 to 9.

H. Fourier Transform Infrared Spectroscopy (FT-IR) Analysis

Precisely 100 mg of potassium bromate (KBr) was weighed with a sensitive weighing balance and blended in with 2.1 mg of adsorbents powder in a mortar and pestle. The combination was packed in a compressor machine until the sample was compacted. samples were set in a cell prior to fixing it in a Parkin Elmer FT-IR framework BX spectrum and spectra perusing (4000-400 cm^{-1}) was taken (Jabar and Odusote, 2020)

I. Scanning Electron Microscopy/ Energy Dispersive X-ray Spectroscopy (SEM/EDX) Analysis

The morphology of the samples and elemental composition of modified and unmodified GS, ES, CS were gotten by Scanning electron microscopy (SEM) utilizing a JSM-7610F (Tokyo, Japan). The equipment is a super high resolution schotty Field emission examining electron microscopy combined with energy dispersive x-beam/x-beam fluorescence spectrometer. The adsorbent surface was considered with magnifying lens worked at 10.0 kV. The samples were covered with a 10 nm thick layer of gold (Munagapati *et al*., 2018; Jabar *et al*., 2020).

IV. DETERMINATION OF QUALITY PARAMETERS IN VEGETABLE OIL

A. Peroxide Value

The peroxide value estimated by dissolving 5 g of the sample in 30 cm^3 of glacia acetic acidic and chloroform combination (3:2 v/v), then, at that point, 0.5 cm^3 of KI was added. The solution was then titrated with standard sodium thiosulphate utilizing starch indicator. Peroxide value was determined utilizing the condition.

Peroxide value (m_{eq}/kg) = $\frac{(S-B)\ X\ MX1000}{W}$ 10

B. Colour and odour

The colour of the oil samples was evaluated by visual examination while the scent of the oil sample was resolved utilizing a glass stoppered bottle flushed with 4 M HCl inside and remotely and washed with refined water. The container was half loaded up with the oil samples and shaken overwhelmingly for around 2 minutes. The stopper was taken out and odour observed by putting nostrils close to the mouth of the bottle (Onawumi *et al*., 2017).

C. Free fatty acid value

The oil samples (10 g) were taken into a spotless and dry conical flask to which 25 ml neutralized 95 % ethanol was added and very much blended to break up the oil in ethanol. Phenolphthalein was added as an indicator. Content of flask was titrated against 0.1N KOH solution. Shaking continually until a pink colour persevered for 30 minutes. The level of free fatty acid (FFA) was determined as (Onawumi *et al*., 2017):

$$FFA(\%) = \frac{VxNx28.2}{W} \quad 11$$

Where:

V = Titre value
N = Normality of the KOH
W = Weight of the oil samples

V. RESULTS AND DISCUSSION

Table 1 shows the physicochemical study carried out on the biomass (PESA). The pH, moistness content (MC), vaporizable material (VM), bulk density (BD), ash content (AC), and fixed carbon (FC) were within the range of those found in literature (Onawumi *et al*., 2021; Sangoremi *et al*., 2024). The prepared GSAC possesses high fixed carbon content and surface area (SA) that boost good adsorption. A good activated carbon should possess a SA of 500-1500 m^2/g (Sangoremi *et al*., 2024).

Table 1: Physicochemical Properties and Proximate Compositions of Modified Egg Shell Adsorbent (ESAC)

S/no	Parameters	Mean ± SE
1	pH	7.80 ± 0.011
2	Moisture content (%)	12.90 ± 0.110
3	Volatile matters (%)	9.90 ± 0.012
4	Ash content (%)	5.80 ± 0.111
5	Fixed carbons (%)	71.40 ± 0.001
6	Bulk density (g/cm^3)	1.33 ± 0.000
7	Surface area (m^2/g)	800.00 ± 0.000

The exterior functional groups determination by FTIR for biosorbent is revealed in Figure 1. The FTIR spectra of modified eggshell adsorbent (MESA) show certain adsorption peaks that are indicative of functional groups. The spectra indicate that the adsorbent has potential adsorption sites as represented by the functional groups like: OH, N-H, C≡N, C=O, CH_3, C-H, and C-Cl which influence the surface chemistry of MESA. The band at 3865.48 cm^{-1} is ascribed to non-bounded OH stretch, the signal at 3441.12 and 3362.24 cm^{-1} proposed N-H stretching, while the signal at 2345.52 and 1698.70 cm^{-1} suggested the C≡N and C=O functional groups, the band at 1450.52 and 1003.02 cm^{-1} showed that of CH_3 and –CH respectively, while the peak at 933.58 cm^{-1} is attributed to C-Cl functional group. Other authors got similar results (Munagapati *et al*., 2018).

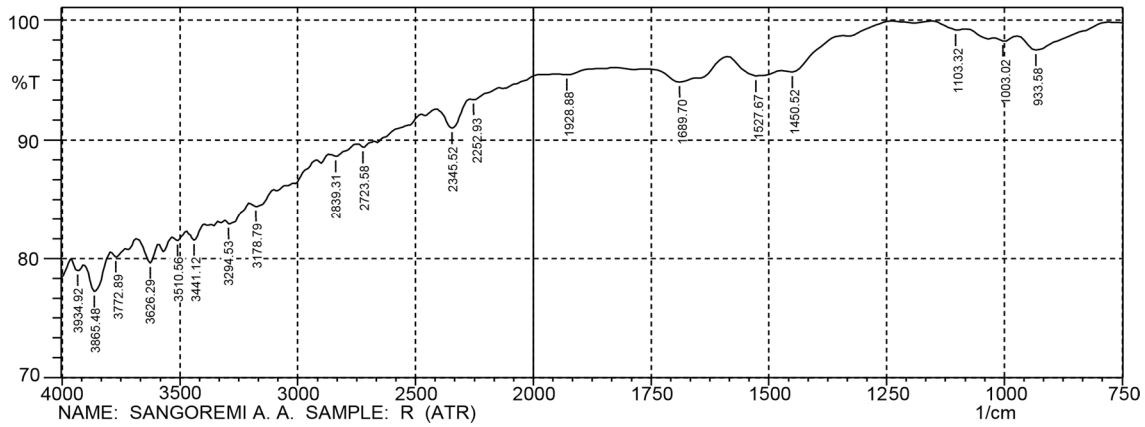


Figure 1: FTIR Analysis of Modified Eggshell Adsorbent

Table 2: The FTIR Spectrum of Modified Eggshell

S.No	Wave number (cm^{-1})	Frequency range (cm^{-1})	Functional group assignment
1	3865.48	> 3500	O-H stretch
2	3441.12	3500-3300	N-H stretch
3	3362.29	3550-3060	N-H stretch
4	2345.52	3300-2500	C≡N stretch
5	1689.70	1715-1680	C=O stretch
6	1450.52	1510-1450	CH_3
7	1003.02	1250-1000 (m-s)	C-H, alkanes and alkylhalides
8	933.58	1000-665 (s)	CCl

Figure 2 reveals the scanning electron micrograph (SEM) of MESA. The surface morphology revealed that the volatiles were released within the microstructure and after acid treatment with H_3PO_4, several pores were formed as seen in the micrograph. This is evidence that activation of biosorbent with orthophosphoric acid promotes better porosity, high carbon content and improved surface reactivity. In addition, the presence of element like carbon, calcium, silicon, calcium, phosphorus and aluminum, iron and sulphur in percentage weights was revealed by EDX (Figure 3) (Onawumi *et al*., 2021; Sangoremi *et al*., 2024)

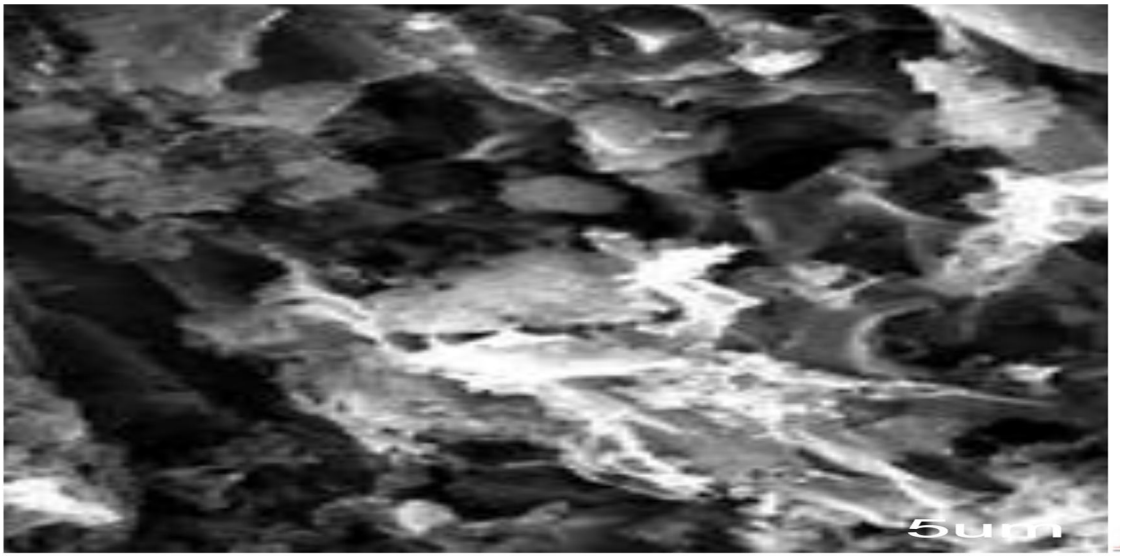


Figure 2: SEM analysis of Modified Eggshell Adsorbent

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Table 4.16: Elemental Composition of Modified Egg Shell Adsorbent

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	93.67	84.43
20	Ca	Calcium	2.41	5.63
15	P	Phosphorus	1.15	1.99
19	K	Potassium	0.65	1.98
14	Si	Silicon	0.81	1.71
13	Al	Aluminum	0.58	1.18
12	Mg	Magnesium	0.32	0.58
26	Fe	Iron	0.12	0.49
16	S	Sulfur	0.17	0.41
17	Cl	Chlorine	0.13	0.35
22	Ti	Titanium	0.00	0.00

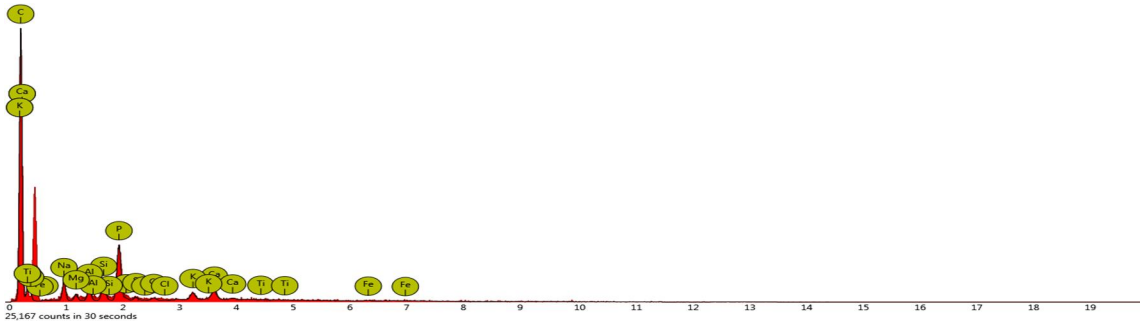


Figure 3: EDX Spectrum of Modified Egg Shell Adsorbent

A. Effects of Adsorbent Dosage on Recovery Efficiency

The dependence of percentage removal (% R) of PO and FFA on the amount of MESA is represented in Figure 4. It was observed that the recovery efficiency of PO and FFA from SVOs improved with a rise in adsorbent dosage of 4 to 12 g until a marginal decrease sets in. The opinion could be ascribed to the accessibility of extra sites and increasing pore surface areas of the adsorbent. This further encourages easier penetration of PO and FFA molecules from SVOs onto the MESA adsorption sites. UShedo *et al.* (2022) made similar observations in their work when FFA was removed from waste cooking oil (WCO). In the current study, the maximum % adsorption at optimum dosage of 12 g was (91.63 and 87.11 %) of PO and FFA respectively. This was in agreement with works of Susilowati *et al.* (2019) and Bavaresco *et al.* (2021) but less than results got by Ushedo *et al.* (2022) when reeds activated carbon (RAC), activated coal ash (ACA), modified corn cob (CCC), and NPD were used to remove FFA from SVOs.

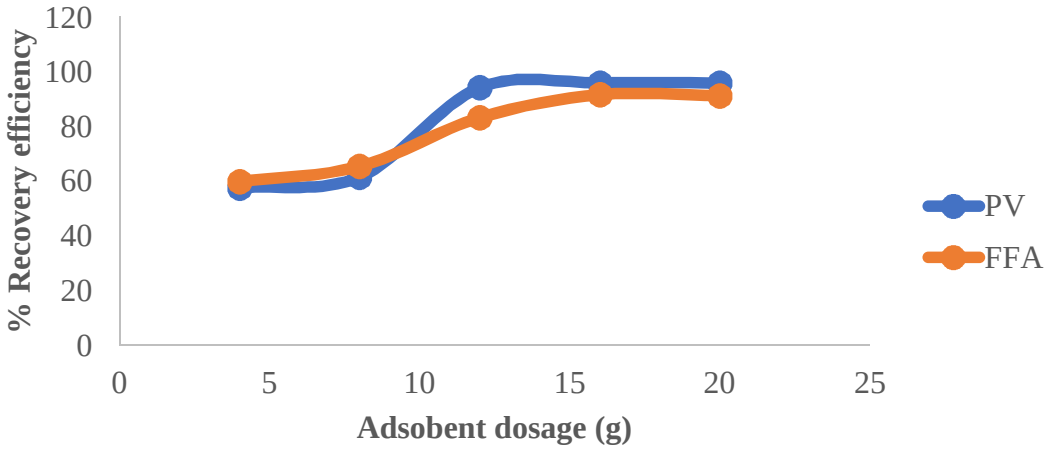


Figure 4: Plots of Adsorbent Dosage and Percentage Recovery of PV and FFA from SVOs using MESA

B. Effects of dosage on colour and odour

The effect of adsorbent dosage was also conducted on colour and odour. Colour and odour are measure of oil quality. UFVO analyzed had dark brown colour and an objectionable odour which may be due to the formation of polymeric compounds resulting from excessive heat which tends to induce colour change (Didar, 2017). Application of adsorbents (unmodified and modified) at adsorbent dosage range of 4g - 20 g were able to remove the objectionable odour to mildly unobjectionable from 4-8 g, and completely unobjectionable from 12-20 g. Consequently, the colour was able to change from dark brown to golden brown upon the application of 4-12 g and to near pale yellow (original colour) from (12-20 g). Similar observations were reported by other researchers; Bavaresco *et al.* (2019) in their work, were able to remove both colour and odour from UFVO using eggshell activated carbon and modified corn cob (CCC), and Onawumi *et al.* (2017) were able to remove both colour and odour from used cooking oil using unmodified eggshell and maize cob as adsorbents. The bleaching ability and odour removal capacity of active carbon were reported by other authors, Didar *et al.* (2017) successfully decolourized and deodourized waste cooking groundnut oil using activated carbon prepared from coconut shell.

VI. CONCLUSION

The MESA possesses high fixed carbon content and surface areas together with well-developed pore structures. The presence of functional groups like OH, N-H, C≡N, C=O, CH_3 and CH boosts its good surface chemistry for adsorption of FFA and PV from SVOs. The adsorption cycle was reliant upon dosage at the optimum condition of 12 g until a marginal decrease sets in.

VII. ACKNOWLEDGMENTS

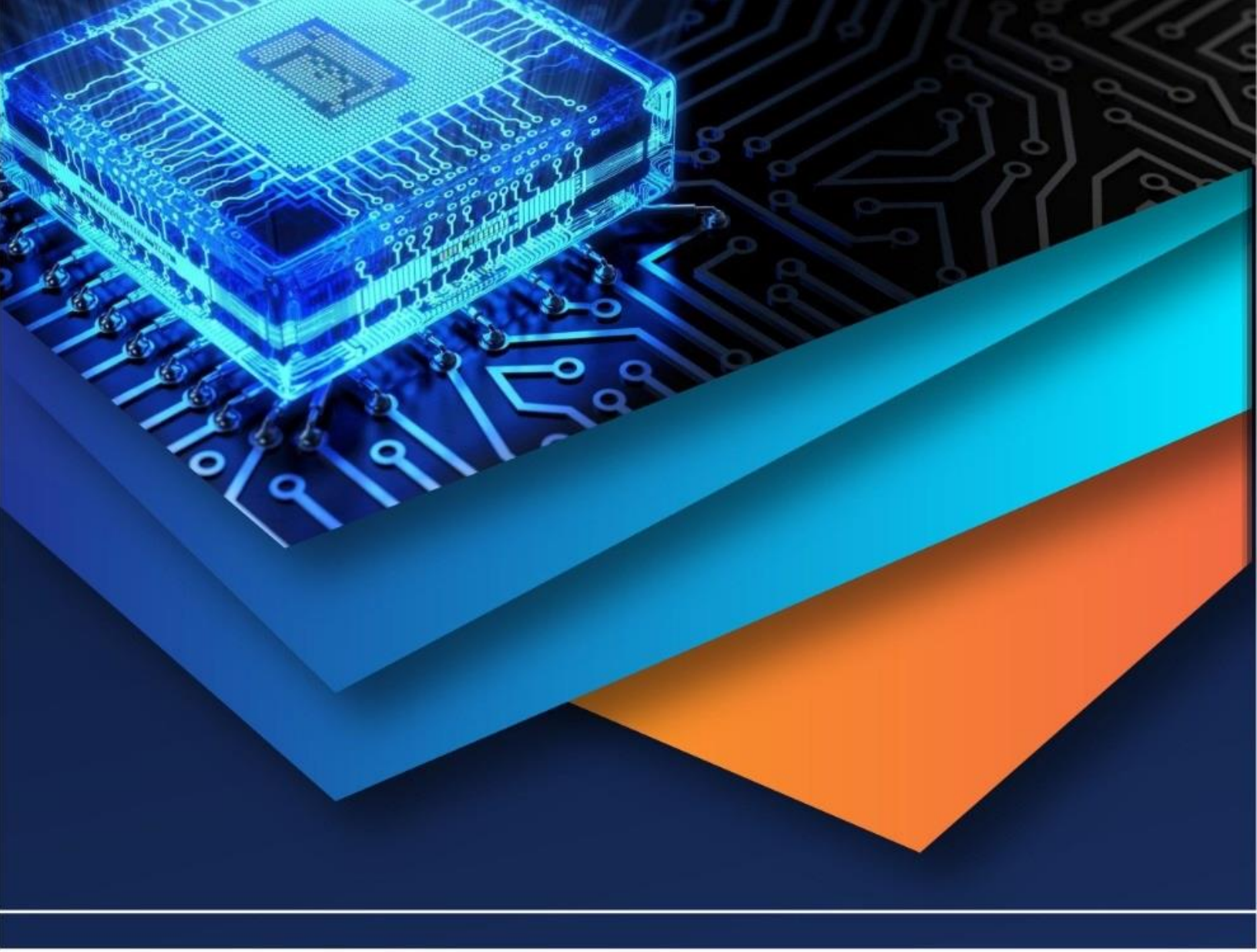
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Conflict of interest: The authors declare that there is no conflict of interest.

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