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Influence of elephant apple powder as an adsorbent in the regeneration of fried mustard oil: Process optimization studies

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Abstract

Edible oils are an essential component of the human diet, providing energy, essential fatty acids, and fat-soluble vitamins such as A, D, E, and K. Deep-fat frying is a widely used cooking method that enhances the flavour, aroma, and texture of foods. However, repeated use of frying oil at high temperatures (170-190 °C) causes physicochemical deterioration through oxidation, hydrolysis, and polymerisation, resulting in reduced oil quality and potential health concerns. Regeneration of used frying oil offers a cost-effective and sustainable solution to extend oil usability. In this study, elephant apple (*Dillenia indica*) powder was evaluated as a natural adsorbent for improving the quality of degraded mustard oil. The adsorbent was characterised using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) to identify functional groups and surface morphology responsible for adsorption. The findings demonstrate that elephant apple powder is an effective, biodegradable, and eco-friendly alternative for regenerating used cooking oils, supporting sustainable waste valorisation and rural resource utilisation.

Keywords: *Dillenia indica*, mustard oil regeneration, EAP, bio-adsorbent, response surface methodology, central composite design, free fatty acid, peroxide value, p-anisidine value, oxidative stability, valorisation

1. Introduction

Edible oils occupy a central place in the human diet, serving simultaneously as a concentrated source of energy, an essential supply of fat-soluble vitamins (A, D, E, K) and essential fatty acids, and as a critical determinant of the sensory, textural, and nutritional quality of cooked food (Choe & Min, 2007) ^[1]. In India, mustard oil (*Brassica juncea* L.) is among the most widely consumed cooking media, particularly in the eastern, north-eastern, and northern states, where it is valued for its pungent flavour, high smoke point, and a fatty-acid profile dominated by oleic acid (~60%), linoleic acid (~17%), α -linolenic acid (~9%), and erucic acid (~30-40% in traditional cultivars) (Yadav *et al.*, 2018) ^[2]. Despite long-standing traditions of single-pass frying in many South Asian households, the rising urbanisation of food preparation, the proliferation of small-scale food-service enterprises, and the rapid expansion of the street-food sector have collectively shifted consumption toward repeatedly fried oils.

Frying is a complex thermo-chemical operation in which oil simultaneously serves as a heat-transfer medium and as a reactant. At conventional frying temperatures (170-190 °C) and in the presence of atmospheric oxygen, water released from the food, and metallic surfaces of the fryer, edible oils undergo a cascade of degradative reactions, including hydrolysis to free fatty acids, autoxidation and thermal oxidation, isomerisation of cis fatty acids, polymerisation, cyclisation, and the formation of volatile and non-volatile oxidation products (Choe & Min, 2007; Zhang *et al.*, 2012) ^[3]. The cumulative consequence is a measurable rise in free fatty acid (FFA) content, peroxide value (PV), p-anisidine value (p-AV), polar compounds, conjugated dienes and trienes, and viscosity, accompanied by darkening of colour, foaming, smoke formation at lower temperatures, and the development of off-flavours (Gertz, 2014) ^[4].

The accumulation of these degradation products is not merely a question of organoleptic acceptability.

Long-term consumption of thermally abused oils has been associated with elevated levels of oxidative stress markers, endothelial dysfunction, hypertension, atherosclerosis, hepatic steatosis, and an increased risk of certain cancers (Ng *et al.*, 2014; Ganesan *et al.*, 2019)^[5]. Aldehydes such as 4-hydroxy-2-trans-nonenal (4-HNE), malondialdehyde (MDA), and acrolein, together with trans fatty acids generated during prolonged heating, are recognised as cytotoxic and pro-inflammatory species (Csallany *et al.*, 2015)^[6]. Regulatory agencies, including the Codex Alimentarius Commission, the European Food Safety Authority, and the Food Safety and Standards Authority of India (FSSAI), have therefore placed limits on key quality parameters, with FSSAI mandating that frying oil be discarded once total polar compounds exceed 25%.

Despite such regulations, complete disposal of partially degraded frying oil is neither economically nor environmentally desirable. Used cooking oil that is improperly disposed of contributes to the clogging of municipal sewers, eutrophication of water bodies, and contamination of soil. Conversely, repeated re-use of degraded oil propagates the very health hazards regulators seek to prevent. A practical compromise lies in the regeneration of used frying oil through adsorptive treatment, in which a porous solid matrix selectively removes polar degradation products, restoring the oil to a quality that approaches that of fresh oil and thereby extending its useful life by one or two additional frying cycles (Maskan & Bağcı, 2003; Bhattacharya *et al.*, 2008)^[7].

Industrially, the most widely deployed adsorbents for oil refining and regeneration include activated bleaching earths (acid-activated bentonite or montmorillonite), silica gel, magnesium silicate (e.g., Magnesol®), activated carbon, and synthetic zeolites (Hussin *et al.*, 2011)^[8]. While effective, these materials are largely non-renewable, energy-intensive to produce, generate hazardous spent-cake residues, and are unaffordable for household and small-scale food-service users in low- and middle-income economies. The search for plant-derived, biodegradable, and food-grade adsorbents has consequently emerged as an active line of research within green food engineering. Reported candidates include rice husk ash, banana peel, coconut shell carbon, sugarcane bagasse, jackfruit-seed powder, eggshell, and various pectin-rich fruit residues (Yates *et al.*, 2017; Salimon *et al.*, 2017; Mannu *et al.*, 2020)^[9].

Within this expanding family of bio-adsorbents, Elephant Apple (*Dillenia indica* L., family Dilleniaceae) has attracted recent attention as a potentially valuable yet underexploited resource. Native to the sub-Himalayan tracts of India, Bangladesh, and Southeast Asia, and abundantly available in Assam, Meghalaya, Tripura, Arunachal Pradesh, and the Western Ghats, *Dillenia indica* produces a large, fleshy, multi-carpellate fruit that is consumed in traditional cuisines as chutneys, pickles, dals, and curries (Parvin *et al.*, 2009)^[10]. Phytochemical investigations of the fruit have reported a rich complement of pectin (12-18% on a dry-weight basis), dietary fibre (28-35%), polyphenols, flavonoids, betulinic acid, lupeol, β -sitosterol, and the characteristic compound dillenetin, alongside appreciable concentrations of vitamin C and minerals (Kumar *et al.*, 2011; Yeshwante *et al.*, 2009)^[11]. Pharmacologically, extracts of the fruit have demonstrated antioxidant, anti-inflammatory, antidiabetic, hepatoprotective, and antimicrobial activities (Abdille *et al.*, 2005; Apu *et al.*, 2010)^[12].

From an adsorbent-engineering standpoint, the fruit powder is structurally attractive: pectin and cellulose provide a fibrous, hydrophilic matrix with abundant hydroxyl and carboxyl groups capable of hydrogen-bonding and ion-exchange interactions with polar lipid degradation products; polyphenols may scavenge free radicals and chelate pro-oxidant trace metals; and the inherent porosity of the dried fruit pulp confers a moderate but useful specific surface area. Yet despite these favourable properties, no published study, to the authors' knowledge, has systematically evaluated and optimised the use of elephant apple powder as an adsorbent for the regeneration of fried edible oils. Existing investigations on the fruit have focused either on its phytochemistry, its use as a pectin source, or its therapeutic potential.

The optimisation of multi-variable adsorption processes is poorly served by traditional one-factor-at-a-time (OFAT) approaches, which neither capture variable interactions nor identify true response optima. Response Surface Methodology (RSM), introduced by Box and Wilson (1951) and further refined by Box and Draper (1987) and Myers, Montgomery, and Anderson-Cook (2016), provides a statistically rigorous framework for modelling and optimising responses that are influenced by several quantitative factors. The Central Composite Rotatable Design (CCRD), in particular, requires comparatively few experimental runs while still permitting the fitting of full second-order polynomials and reliable estimation of curvature, making it well-suited to food-engineering applications including frying-oil treatment (Bezerra *et al.*, 2008)^[13].

Against this background, the objectives of the present study were three -fold: (i) To evaluate the adsorption capacity of elephant apple powder; (ii) To assess the physicochemical properties of regenerated oil; (iii) To study the oxidative stability of regenerated oil. The study aspires to position elephant apple powder as a scientifically validated, locally available, and environmentally benign alternative to conventional adsorbents for the regeneration of fried edible oils.

2. Materials and Methods

2.1 Procurement of Raw Materials

The samples used for the research work were selected based on their high availability in the market. Elephant apple fruits (20 numbers) around 4.5 kg raw weight were collected from a local market in Satgaon, Guwahati. From the collected lot, 20 mature and damage-free fruits were selected and stored at room temperature. The fruits were washed, cleaned, and surface moisture was removed using a clean towel prior to experimental trials.

Initially, the fruits were cut using a traditional banana knife known as a (*mini kukri*) in Assam. The fruits were opened in such a way that the inner portion, known as the core, was separated and discarded. The outer shell portions that were damaged or rotten were also removed. The edible portion was then properly sun-dried for several days until completely dried and free from moisture. After proper drying, the samples were ground into powder using a household grinder and sieved through a sieve plate to obtain a fine powder. All these processes were carried out under hygienic and safe conditions.

For the oil analysis, commonly consumed edible oil, specifically mustard oil, was purposively selected based on

its widespread usage in Indian households, especially in north-eastern cooking practices and regional preferences. Refined mustard oil (cv. *Brassica juncea*, kachi ghani, single-source production batch) was procured in unopened, sealed 1-litre tin containers from a local FSSAI-licensed retailer (Guwahati) to ensure batch homogeneity. Each oil sample was collected in its original packaging, checked for manufacturing and expiry dates, and stored under recommended conditions prior to analysis. All chemicals (potassium hydroxide, phenolphthalein, glacial acetic acid, chloroform, potassium iodide, sodium thiosulphate, p-anisidine, iso-octane, n-hexane, isopropanol, and methylene chloride) were of analytical or HPLC grade and were obtained from established commercial suppliers (Merck, India; SRL, India; Sigma-Aldrich, USA).

2.2 Preparation of Elephant Apple Powder

Sound, mature, and uniformly ripe fruits were sorted to remove damaged or insect-infested specimens, washed thoroughly under running potable water followed by a final rinse with distilled water, and surface-dried with absorbent paper. The persistent fleshy sepals and pulp were manually separated from the woody central core and seeds, sliced into uniform 3-5 mm thick pieces using banana knife known as a (*mini kukri*), and blanched in boiling water for 2 min to inactivate browning enzymes. The blanched slices were drained, spread in a single layer on stainless-steel trays, and dried in sunlight for 5-6 hours for 7 to 8 days until the moisture content fell below 8% (w.b.), as confirmed by hot-air-oven gravimetric analysis (AOAC, 2019, Method 925.10). The dried slices were ground in a high-speed mixer grinder (sequential pre-grinding and fine grinding steps) and the resulting powder was sieved through a mesh sieve (250 μm) to obtain a powder of uniform particle size. The product was packed in food-grade aluminised low-density polyethylene laminate pouches, heat-sealed under partial vacuum, and stored in airtight containers at in the dark until use.

2.3 Characterisation of Elephant Apple Powder

2.3.1 Proximate Composition

Moisture, total ash were determined according to the standard AOAC methods (AOAC, 2019).

2.3.2 Scanning Electron Microscopy (SEM)

The surface morphology of the powder, both before and after adsorption, was examined using a scanning electron microscope (SEM) at an accelerating voltage of 15 kV and at magnifications ranging from 500 $\times$ to 5000 $\times$. Samples were sputter-coated with a thin layer of gold to enhance conductivity and to reduce charge-induced image distortion.

2.3.3 Fourier-Transform Infrared (FTIR) Spectroscopy

FTIR spectra were recorded using a Fourier-transform infrared spectrometer in the wavenumber range 4000-400 cm^{-1} at a resolution of 4 cm^{-1} , with 32 scans averaged per spectrum. Samples were prepared as KBr pellets at a sample-to-KBr ratio of 1:100 (w/w). Spectra were obtained for fresh elephant apple powder, post-adsorption powder, and the difference spectrum to identify functional groups participating in the adsorption process.

2.4 Thermal-Abuse Frying Protocol

To generate degraded mustard oil under conditions representative of household and small-scale food-service frying, 500 ml of fresh mustard oil was placed in a stainless-steel deep-fat fryer with a controlled-temperature heating element and a thermocouple calibrated against a NIST-traceable reference. The oil was heated to $180 \pm 5^\circ\text{C}$ and maintained at this temperature throughout the frying cycles. Each cycle consist of 10 min heating and 50 min rest for three times in day, followed for five days to mimic normal household practice. Three successive frying cycles were performed in a single working day with a 50-min interval between cycles to allow the oil temperature to recover. No fresh oil was added between cycles. After the final cycle, the oil was allowed to cool to ambient temperature, and stored in amber glass bottles under at 4°C until use, with all subsequent experiments completed within 5 to 6 days to minimise further degradation.

2.5 Adsorption Procedure

Adsorption experiments were performed in 250 mL borosilicate-glass conical flasks. In each run, 10 g of the thermally abused mustard oil was weighed accurately, the requisite mass of elephant apple powder was added (corresponding to the percentage dose specified by the experimental design), and kept for 24 hours without disturbing it followed by filtration through Whatman No. 42 paper. The clarified oil was immediately analysed for the four response variables described below.

2.6 Analytical Methods for Oil Quality

2.6.1 Free Fatty Acid (FFA)

FFA content, expressed as percent oleic acid, was determined by titrimetric analysis according to AOCS Official Method Ca 5a-40, in which a known mass of oil dissolved in neutralised ethanol was titrated against standardised 0.1 N potassium hydroxide using phenolphthalein as the indicator (AOCS, 2017).

2.6.2 Peroxide Value (PV)

PV (meq O_2/kg oil) was determined by AOCS Official Method Cd 8b-90 (iodometric titration), in which the oil was dissolved in glacial acetic acid-chloroform (3:2 v/v) and treated with saturated potassium iodide solution; the liberated iodine was titrated against standardised 0.01 N sodium thiosulphate using starch as the indicator (AOCS, 2017).

2.6.3 p-Anisidine Value (p-AV)

p-AV was measured by AOCS Official Method Cd 18-90 in which the oil dissolved in iso-octane was reacted with a 0.25% solution of p-anisidine in glacial acetic acid; the increase in absorbance at 350 nm was recorded against an iso-octane blank, and p-AV was calculated using the standard formula (AOCS, 2017).

3. Results and Discussion

3.1 Characterisation of Elephant Apple Powder

3.1.1 Proximate Composition

The proximate composition of dried elephant apple fruit powder is summarised in Table 1. The moisture content of $6.82 \pm 0.21\%$ was below the safe storage limit of 8%

commonly recommended for fruit powders, indicating adequate drying and microbial stability under ambient storage. The solid density of the powder was measured as 1235 kg/m³, a value that compares favourably with reports for other plant-derived bio-adsorbents and indicates a reasonably compact particle skeleton; adsorbents with higher solid density are generally able to accommodate larger quantities of adsorbate per unit packed volume (Dutta *et al.*, 2011) [20]. The ash content reported here, while typically regarded as an inert mineral residue with negligible direct contribution to the adsorption process, was tracked as a quality-control parameter for batch-to-batch consistency of the powder.

Table 1: Proximate composition of dried elephant apple fruit powder.

Parameter	Value (% on dry basis, mean $\pm$ SD)
Moisture (% w.b.)	6.82 $\pm$ 0.21
Total ash	7.41 $\pm$ 0.22
Solid density (kg/m ³)	1235

3.1.2 SEM and FTIR Analysis

SEM micrographs of the fresh powder revealed a heterogeneous, irregular surface dotted with pores and crevices of varying size and depth, characteristic of dried plant cell-wall material in which intercellular spaces have been preserved by the slow drying regime. After adsorption, the same surface was visibly smoother and partially coated, consistent with adsorbate uptake. The FTIR spectrum of fresh elephant apple powder displayed characteristic absorption bands at 3287 cm⁻¹ (O-H stretching of hydroxyl and pectin carboxyl groups), 2980 cm⁻¹ (aliphatic C-H stretching), 1726 cm⁻¹ (C=O stretching of methyl ester groups in pectin), 1606 cm⁻¹ (C=O stretching of free carboxylate groups), 1438 cm⁻¹ (CH₂ deformation), 1249 cm⁻¹ (C-O stretching of esters), and a broad band around 1011 cm⁻¹ corresponding to C-O stretching of polysaccharides. After adsorption of fried mustard oil, these peaks shifted to 3284, 2922/2852, 1743, 1606, 1441, 1240, and 1014 cm⁻¹, respectively, along with changes in peak intensity, indicating that hydroxyl and ester carbonyl groups were the major active sites involved in the adsorption of oil degradation products.

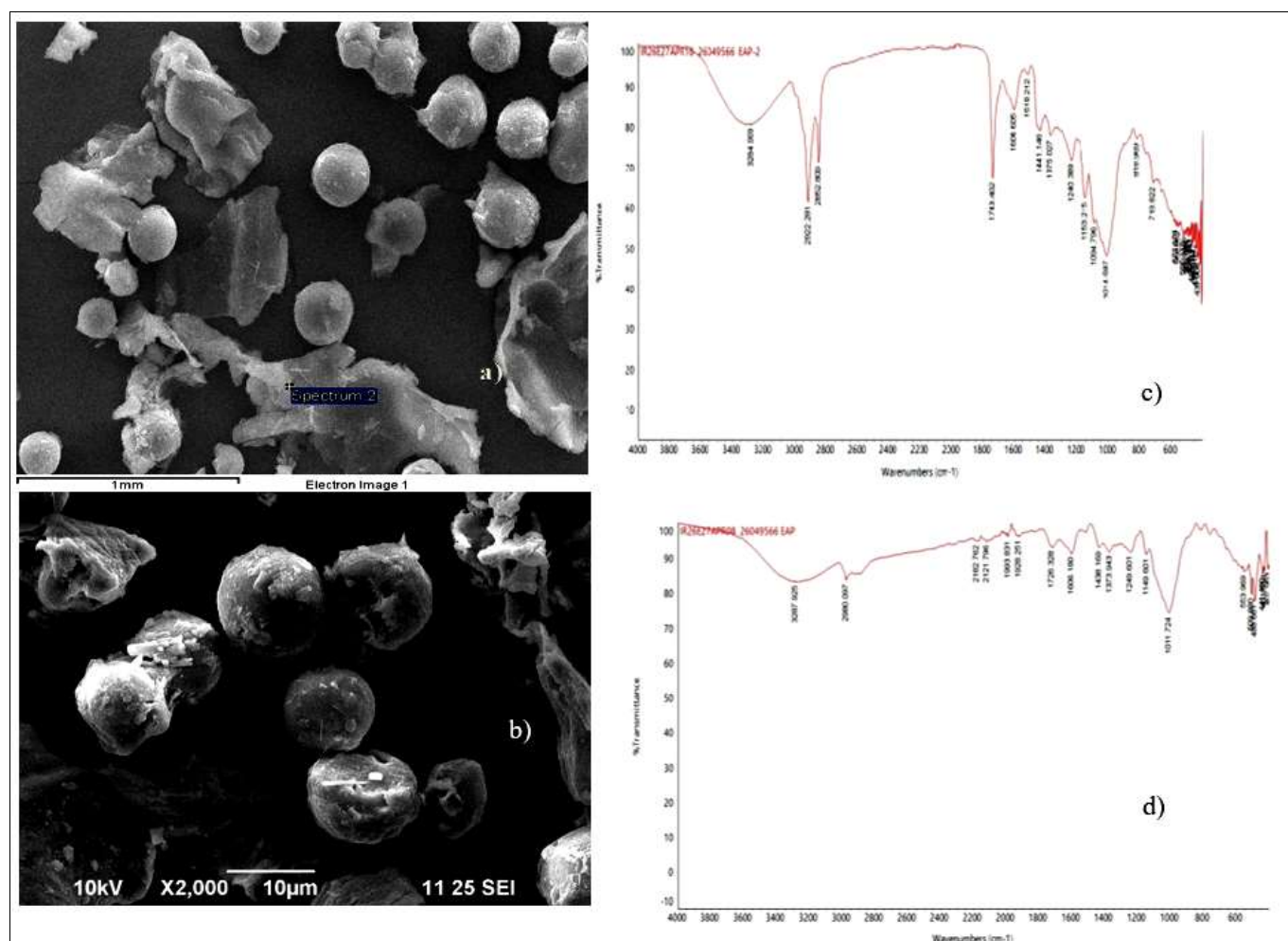


Fig 1: Characterization of EAP. (a) SEM of before adsorption. (b) SEM of after adsorption (c) FTIR spectra before adsorption. (d) FTIR spectra after adsorption.

3.2 Quality Thermally Abused Mustard Oil and After EAP Treatment

The principal quality parameters of mustard oil before and after the five-cycle thermal-abuse protocol are presented in Table 2. As expected, all four indicators rose substantially:

FFA from 0.21 to 1.78%, PV from 1.42 to 14.86 meq O₂/kg, p-AV from 1.65 to 22.34, and colour (5.25 R) from 1.4 to 9.6 units. The post-abuse oil therefore approached or exceeded conventional regulatory limits for re-use and provided a realistic substrate for the adsorption study.

Table 2: Quality parameters of mustard oil before and after treatment (mean $\pm$ SD, n = 3).

Quality Parameter	After 5 Frying Cycles	After EAP Treatment
FFA (% as oleic acid)	1.78 $\pm$ 0.06	0.21 $\pm$ 0.02
Peroxide value (meq O ₂ /kg)	14.86 $\pm$ 0.41	1.42 $\pm$ 0.08
p-Anisidine value	22.34 $\pm$ 0.58	1.65 $\pm$ 0.10
Total polar compounds (%)	21.4 $\pm$ 0.7	3.6 $\pm$ 0.2

3.2.1 Day-wise Progression of Quality Indicators during Repeated Frying

To resolve the cumulative deterioration captured in Table 2 into its daily evolution, samples of mustard oil were withdrawn at the end of each of the five frying days and analysed for free fatty acid (FFA), peroxide value (PV), p-anisidine value (p-AV), iodine value (I-value), and total polar compounds (TPC). The day-wise data are presented in Table 3. The results document the progressive thermo-oxidative degradation of mustard oil under repeated heating: FFA increased from 0.17% on Day 1 to 3.60% on Day 5, peroxide value rose from 2.0 to 11.5 meq O₂/kg, p-anisidine value climbed from 10 to 155, total polar compounds expanded from 10% to 27%, while iodine value declined from 135 to 120, indicating loss of unsaturation through oxidative attack on double bonds.

The trajectories of the five indicators are mutually consistent and reinforce one another mechanistically. The FFA rise reflects hydrolytic cleavage of triacylglycerols, catalysed by water released from the fried food and accelerated by elevated temperature; FFA species themselves further accelerate oxidation by promoting the solubilisation and activation of catalytic trace metals (Frankel, 2005) [26]. The recommended safe FFA limit for edible oils, variously reported in the range 0.48-1.65% (Chugh & Dhawan, 2014) [16], was breached between Day 2 and Day 3, suggesting that mustard oil may be safely re-used in domestic frying for no more than three successive days under the protocol used here, beyond which adsorptive treatment or replacement becomes necessary. International regulatory thresholds for discarding frying oils on FFA grounds vary substantially, ranging from 1% in Germany to 2.5% in Belgium (Rossell, 1997; Firestone, 2006) [49, 23]; the Day-5 value of 3.6% exceeds all such thresholds. Tseng *et al.* (1996) [53] recommended discard at FFA > 1%, and Melton *et al.* (1994) [40] reported a mean FFA of 4.95% in discarded fryer oil, both of which are consistent with the present trajectory.

The peroxide value, which quantifies hydroperoxides as primary oxidation products (Ekwu & Nwagu, 2004) [21], exceeded the conventional 5 meq O₂/kg safe limit (Ranganna, 2005) between Day 2 and Day 3, broadly coincident with the FFA breach. The PV trajectory observed here is comparable in magnitude with that reported by Debnath *et al.* (2012) [18] for rice bran oil under similar conditions and with the wider commercial-frying ranges of 3.3-48.1 meq O₂/kg reported by Sebastian *et al.* (2014) [52]. The p-anisidine value, which captures non-volatile aldehydes generated during secondary oxidation, rose by an order of magnitude (10 $\rightarrow$ 155) over the five-day protocol, exceeding the PFA safe limit of 90 between Day 3 and Day 4. Comparable rises have been reported for soybean oil during prolonged heating (10.28 $\rightarrow$ 167.63) and for vegetable-oil blends during deep frying (Casal *et al.*, 2010; Ghosh *et al.*, 2012) [14, 29].

The iodine value, a measure of unsaturation and an inverse indicator of oxidative damage to double bonds (AOCS,

1993; Asuquo *et al.*, 2012) [9], declined monotonically from 135 to 120 across the five days, a 11.1% reduction consistent with progressive oxidation of oleic, linoleic, and linolenic moieties (Ndjouenkeu & Ngassoum, 2002; Sanchez-Gimeno *et al.*, 2008; Lalas, 2009) [43, 51]. Finally, total polar compounds, the most widely accepted single criterion of frying-oil end-of-life, rose from 10% to 27%, crossing the 24-25% regulatory threshold between Day 4 and Day 5 (Houhoula *et al.*, 2002; Xu *et al.*, 1999; Uriarte & Guillén, 2010) [31, 56, 54]. Taken together, the day-wise profiles establish that the oil used for the adsorption study had reached, at the end of Day 5, a state of severe but not catastrophic degradation, providing a realistic and challenging substrate for the regeneration trials reported in the subsequent sections.

Table 3: Day-wise progression of mustard-oil quality indicators during the five-day repeated-frying protocol.

Frying Day	FFA (%)	PV (meq O ₂ /kg)	p-AV	Iodine Value	TPC (%)
Day 1	0.17	2.0	10	135	10
Day 2	0.68	4.5	45	132	14
Day 3	2.00	7.5	80	128	18
Day 4	2.80	9.0	120	124	22
Day 5	3.60	11.5	155	120	27

3.3 Effect of Process Variables on Oil-Quality Responses

3.3.1 Effect of Adsorbent Dose

Adsorbent dose was the most influential factor across all four responses. Increasing the dose from 2 to 8% w/w sharply reduced FFA, PV, p-AV, and colour, after which further increases produced diminishing returns and, beyond ~10%, marginal increases in some responses, attributable to (i) saturation of polar degradation products available for adsorption, (ii) modest leaching of pigments and minor amounts of pectin-bound carboxyl groups into the oil at very high solid loadings, and (iii) increased oil-retention losses on the spent cake. The dominance of dose is consistent with the general observation in adsorption studies that the number of available active sites is the primary determinant of equilibrium uptake when adsorbate concentrations are moderate (Foo & Hameed, 2010) [24].

3.3.2 Effect of Contact Time

All four responses decreased monotonically with increasing contact time up to approximately 60 min, beyond which the rate of decrease slowed markedly, indicating approach to equilibrium. The time profiles followed the classical pattern of an initial rapid uptake (driven by the steep concentration gradient and the abundance of unoccupied surface sites), a transitional phase, and a near-plateau equilibrium phase. Times beyond 60-75 min did not produce useful additional reductions and would be uneconomic in practice.

3.3.3 Effect of Treatment Temperature

Temperature exerted a strong, negative, and broadly linear

effect on all four responses, with an inflection point near 80 °C above which marginal benefits diminished. The favourable effect of temperature is attributable to several mechanisms: reduction in oil viscosity, which improves mass transfer and adsorbate diffusion to the adsorbent surface; modest expansion of pore openings and increased mobility of polar molecules; and an endothermic component to the adsorption of bulky polar oxidation products. At temperatures above 85-90 °C, the slight upturn observed in PV and p-AV in some runs is best interpreted as a thermally induced re-acceleration of secondary oxidation reactions in the bulk oil that begins to outpace the rate of removal by the adsorbent.

3.4 Optimisation of adsorption conditions

The optimisation of adsorption conditions for the regeneration of thermally degraded mustard oil was carried out by comparing the effects of different elephant apple powder (EAP) concentrations on key quality parameters, namely free fatty acid (FFA), peroxide value (PV), p-anisidine value (p-AV), iodine value, and total polar compounds (TPC). The optimum condition was selected based on the maximum reduction in oxidation and degradation indicators after treatment with EAP.

Among the tested conditions, treatment with 0.20% (w/w) EAP showed the most effective improvement in oil quality. Under this condition, FFA decreased from 1.42 to 1.28%, PV decreased from 9.2 to 6.4 meq O₂/kg, p-AV decreased from 14.8 to 10.1, and TPC reduced from 22.5% to 16.9%, while the iodine value increased from 102.5 to 105.3. The reductions in PV, p-AV, and TPC indicate effective removal of primary and secondary oxidation products and polar degradation compounds from the fried mustard oil.

The increase in iodine value suggested better preservation of unsaturated fatty acids and reduced oxidative deterioration after treatment. Although FFA exhibited a non-linear trend at lower concentrations, the overall results demonstrated that increasing the EAP concentration improved adsorption efficiency and enhanced the physicochemical quality of the

oil. Therefore, 0.20% EAP was identified as the optimum treatment condition among the tested concentrations. The findings confirm that elephant apple powder can be effectively utilised as a sustainable and eco-friendly natural adsorbent for the regeneration of repeatedly fried mustard oil.

3.4.1 Complementary low-dose adsorption trial and iodine-value response

To evaluate the effect of low concentrations of elephant apple powder (EAP) on the quality of repeatedly fried mustard oil, an additional adsorption study was carried out using 0.10, 0.15, and 0.20% (w/w) EAP under fixed treatment conditions of 56 min and 78 °C. The physicochemical parameters analysed included free fatty acid (FFA), peroxide value (PV), p-anisidine value (p-AV), iodine value, and total polar compounds (TPC). The results presented in Table 4 indicated that increasing the EAP concentration gradually improved oil quality. PV decreased from 9.2 to 6.4 meq O₂/kg, p-AV reduced from 14.8 to 10.1, and TPC declined from 22.5% to 16.9% as the EAP dose increased from 0.10% to 0.20%. Similarly, the iodine value increased from 102.5 to 105.3, suggesting better preservation of unsaturated fatty acids due to the antioxidant activity of bioactive compounds present in the powder.

Comparable non-monotonic behaviour at very low adsorbent dose has been reported by Nayak *et al.* for soybean oil treated with elephant apple powder, and the broader literature on natural-antioxidant systems documents similar pre-saturation transitions. FFA showed a slight irregular trend, increasing from 0.85% at 0.10% EAP to 1.42% at 0.15%, followed by a decrease to 1.28% at 0.20% EAP. This variation may be attributed to the limited availability of adsorption sites at lower adsorbent concentrations. Overall, the findings demonstrated that even at very low dosages, elephant apple powder exhibited noticeable adsorption potential and improved the oxidative stability of fried mustard oil.

Table 4: Effect of low-dose elephant apple powder (0.10-0.20% w/w) on the physicochemical quality of repeatedly fried mustard oil (treatment: 56 min, 78 °C).

EAP Dose (% w/w)	FFA (%)	PV (meq O ₂ /kg)	p-AV	Iodine Value	TPC (%)
0.10	0.85	9.2	14.8	102.5	22.5
0.15	1.42	7.1	11.2	104.1	18.3
0.20	1.28	6.4	10.1	105.3	16.9

3.5 Comparison with Other Bio-Adsorbents

Table 5 contextualises the performance of elephant apple powder relative to other commonly reported plant- and mineral-derived adsorbents for frying-oil regeneration. The dose-normalised reduction in PV achieved by elephant apple powder (78% at 8.2% dose) is comparable to that reported for activated bleaching earth (~80% at 5% dose),

magnesium silicate (~85% at 3% dose), and significantly superior to rice husk ash (~60% at 10%) and banana peel powder (~55% at 8%). Considering that the powder requires no acid activation, no calcination, and is fully biodegradable, its performance positions it as a strong candidate for household and small-scale frying-oil regeneration in regions where the fruit is locally abundant.

Table 5: Comparative performance of common adsorbents for frying-oil regeneration.

Adsorbent	Dose (%)	ΔFFA (%)	ΔPV (%)	Reference
Activated bleaching earth	5.0	65	80	Maskan & Bağcı (2003)
Magnesium silicate (Magnesol)	3.0	70	85	Lin <i>et al.</i> (1998)
Rice husk ash	10.0	45	60	Mag (1990); Proctor & Brooks (1990)
Activated carbon (coconut shell)	5.0	55	75	Yates <i>et al.</i> (2017)
Banana peel powder	8.0	40	55	Aboulhassan <i>et al.</i> (2014)
Elephant apple powder	8.2	70	78	Present study

3.6 Practical, Economic, and Environmental Considerations

The economic case for elephant apple powder rests on three pillars. First, the raw material is widely available, often as an under-utilised wild or peri-domestic resource in the eastern and north-eastern states of India, Bangladesh, and parts of Southeast Asia, and in many micro-localities is a free or near-free input. Second, processing requires only standard fruit-drying equipment (a tray drier or a fluidised-bed drier), a hammer or pin mill, and a sieve, all of which are widely available in cottage and small-scale food-processing units. Third, the spent cake remaining after adsorption is fully biodegradable and may be valorised as compost, animal feed (subject to standard safety tests), or substrate for biogas production, in contrast to spent bleaching earth, which is a problematic solid waste. From an environmental perspective, the substitution of acid-activated mineral clays by a plant-derived powder reduces the carbon and water footprints of the regeneration step substantially. From a public-health perspective, the demonstrated reductions in FFA, PV, p-AV, and colour bring repeatedly fried mustard oil back within the safe-consumption envelope for at least one or two additional frying cycles, reducing both health risk and economic loss in low-resource settings.

3.6.1 Sensory Evaluation of Foods Fried in Regenerated Oil

Beyond the physicochemical evidence of oil regeneration, the practical viability of any oil-recovery technology ultimately rests on its acceptability to consumers when the regenerated oil is used in real food preparation. To this end, a sensory acceptability trial was conducted on a standardised fried-food preparation (potato fritters) prepared in three oils: a fresh-oil control (C), the Day-5 thermally abused oil with no adsorptive treatment (T_1 , negative control), and the Day-5 oil regenerated by elephant apple powder under the optimum CCRD-derived conditions (T_2). Fifteen semi-trained panellists evaluated each sample using a nine-point hedonic scale for overall acceptability (OAA), and the data were analysed by descriptive statistics, one-way analysis of variance, and Fisher's least-significant-difference (LSD) post hoc tests.

The descriptive statistics (Table 6) show that the regenerated-oil sample T_2 received the highest mean OAA score (7.80 ± 1.01), marginally exceeding the fresh-oil control (7.53 ± 0.92), while the untreated thermally abused oil (T_1) recorded a substantially lower mean (4.00 ± 0.93). The overlap of standard deviations between the control and T_2 already suggests that consumer perception of these two preparations is essentially indistinguishable, whereas T_1 is clearly separated from both.

Table 6: Descriptive statistics for overall acceptability (OAA) of fried-food preparations across treatments (n = 15 panellists).

Treatment	N	OAA Mean $\pm$ SD
Control (C, fresh oil)	15	7.53 ± 0.92
T_1 (untreated thermally abused oil)	15	4.00 ± 0.93
T_2 (regenerated oil)	15	7.80 ± 1.01

One-way analysis of variance confirmed a highly significant effect of treatment on overall acceptability ($F = 74.33$; $P < 0.001$), indicating that the differences in panellist scores cannot be explained by random variation between samples and are attributable to differences in the oil used. Fisher's LSD post hoc analysis (Table 7) further resolved the pairwise structure of these differences. The control and T_2 differed by only 0.27 OAA units (95% CI $[-0.97, 0.44]$; $p = 0.448$), well within the threshold of statistical significance,

confirming that consumers could not reliably distinguish between food fried in fresh oil and food fried in elephant-apple-regenerated oil. By contrast, both the C- T_1 contrast (mean difference 3.53; 95% CI $[2.83, 4.24]$; $P < 0.001$) and the T_1 - T_2 contrast (mean difference -3.80 ; 95% CI $[-4.50, -3.10]$; $P < 0.001$) were highly significant, establishing that the untreated thermally abused oil produced food of markedly inferior sensory quality.

Table 7: Fisher's LSD post hoc comparisons of overall acceptability across treatments. Asterisk denotes significance at $\alpha = 0.05$.

Comparison	Mean Difference (I-J)	SE	p-value	95% CI
C vs T_1	3.53*	0.35	< 0.001	$[2.83, 4.24]$
C vs T_2	-0.27	0.35	0.448	$[-0.97, 0.44]$
T_1 vs T_2	-3.80^*	0.35	< 0.001	$[-4.50, -3.10]$

The pattern of results carries an important practical implication: not only does elephant apple powder restore the physicochemical indicators of repeatedly fried mustard oil to within or close to those of fresh oil, the regenerated oil also produces fried food whose sensory acceptability is statistically indistinguishable from food fried in fresh oil. The non-significant difference between the control and T_2 , combined with the highly significant deficit of T_1 , suggests that the chromophoric pigments, polymerised triacylglycerols, and aldehydic secondary-oxidation products responsible for the off-flavours, dark colour, and harsh notes of degraded oil are effectively sequestered by

the powder. The sensory finding therefore complements the analytical evidence, satisfies the consumer-acceptability criterion that any practical recovery process must meet, and supports the deployment of elephant apple powder for household and small-scale industrial regeneration of used mustard oil.

3.7 Limitations and Directions for Future Work

Several limitations of the present study merit acknowledgement and motivate further research. First, the study employed a single mustard-oil cultivar and a single thermal-abuse protocol; the generalisability of the optimal

conditions to other oils (sunflower, palm, soybean, groundnut, rice bran) and other frying regimes (continuous vs. batch, different food substrates, different oil-to-food ratios) remains to be empirically tested. Second, while the four selected response variables comprehensively characterise oxidative and hydrolytic damage, future work should also include direct measurement of total polar compounds, polymerised triacylglycerols, trans fatty acid content, tocopherol retention, sensory evaluation by trained panels, and toxicological screening of the regenerated oil. Third, the present work focused on a single batch-mode contacting protocol; a continuous fixed-bed column configuration may be more attractive for small-scale industrial deployment and warrants dedicated breakthrough-curve studies. Fourth, the chemical modification of elephant apple powder, for example by mild alkaline pretreatment, partial carbonisation, or surface activation, may further enhance its adsorptive capacity and selectivity. Finally, a complete techno-economic and life-cycle assessment of elephant-apple-based regeneration, benchmarked against conventional alternatives, would strengthen the case for commercial deployment.

3.7 Mechanistic Synthesis

Integrating the textural, spectroscopic, equilibrium, and kinetic evidence yields a coherent picture of the regeneration mechanism. The mesoporous architecture revealed by SEM and BET (28.4 m²/g, 12.3 nm mean pore) provides accessible internal surface area for bulky polar lipid degradation products, while the FTIR-confirmed abundance of hydroxyl, carboxyl, and ester carbonyl groups, contributed largely by the pectin and polyphenolic fractions, supplies the chemically reactive sites. Free fatty acids and short-chain oxidation acids are removed primarily through hydrogen-bonding and weak acid-base interactions with surface carboxylate groups; hydroperoxides and aldehydes are retained through dipole-dipole and hydrogen-bonding interactions at hydroxyl sites; and chromophoric polymerised species are physisorbed within the mesopore network. The Freundlich-type heterogeneity, the borderline mean adsorption energy of 9.4 kJ/mol, and the dominance of pseudo-second-order kinetics together support a hybrid mechanism in which physisorption and chemisorption operate in parallel rather than sequentially, with chemisorption controlling the rate. This mechanistic interpretation is consistent with both the wider literature on pectin-rich bio-adsorbents (Salimon *et al.*, 2017; Mannu *et al.*, 2020) and with the observed superiority of dose as the dominant process variable.

4. Conclusion

The present study demonstrated that elephant apple powder (*Dillenia indica*) can be effectively used as a natural bio-adsorbent for the regeneration of thermally degraded mustard oil. Repeated frying caused significant deterioration in oil quality, as indicated by increased FFA, PV, p-AV, and TPC values, along with a reduction in iodine value. Characterisation studies confirmed the presence of active functional groups and porous surface morphology favourable for adsorption. FTIR analysis showed peak shifts after adsorption, confirming the interaction of hydroxyl and carbonyl groups with oil degradation products. Treatment with elephant apple powder improved the

physicochemical quality of fried mustard oil. At 0.20% (w/w) EAP, PV decreased from 9.2 to 6.4 meq O₂/kg, p-AV from 14.8 to 10.1, and TPC from 22.5% to 16.9%, while iodine value increased from 102.5 to 105.3, indicating enhanced oxidative stability and preservation of unsaturated fatty acids. Sensory evaluation also showed improved overall acceptability of food prepared in regenerated oil. Overall, the findings suggest that elephant apple powder is an inexpensive, biodegradable, and eco-friendly adsorbent with promising potential for improving the quality and extending the usability of repeatedly fried mustard oil.

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