

## Highly Efficient Clay Based Degumming Aid for Phosphorous Removal from Crude Palm Oil

Indu Bajpai\*, Pawan Pal, Sanjeev Bhasin

Innovation and Knowledge Center, Ashapura Minechem Limited, Madhapar, Bhuj, Kutch, Gujarat, 370020, India

### ABSTRACT

The removal of phosphorous from crude oil is a complex and necessary step to enhance the oxidative stability and shelf life of edible oil. In this aspect, the current work proposed clay based degumming aid for the removal of phosphorous from the crude palm oil during the bleaching process. The state of art qualitative and quantitative techniques was adopted for the study of physical and chemical properties of different raw materials and oil. A profound study on the stability of the degumming aid is based on the assessment of phosphorous removal efficiency with time (up to 6 months) and bleachability. The results of current work revealed that clay based degumming aid is highly effective in removing phosphorous up to 96%. Additionally, the bleaching performance of clay-based aid is also palpable (around 45%), which influences the deodorization process of the crude palm oil positively. The clay based degumming aid eliminate separate degumming process with acids, which directly impact the oil processing time and cost. Overall, proposed clay based degumming aid is highly efficient, stable for long time and cost effective as well, for the removal of undesired phosphorous from the crude palm oil during the bleaching process.

**Keywords:** Bleaching, clay minerals, deodorization, edible oil

### INTRODUCTION

The market of edible oil is very wide and numbers of vegetable oils are produced, transported and consumed everywhere in the world. The global market of palm oil was reported to grow from 88.8 million tons to 111 million tons with the rate of 5% in the coming three years (Majiz *et al.* 2021). Indonesia, India and China are the top three palm oil consumer countries. Interestingly, Indonesia and Malaysia are the two major countries which produce

around 85% of the world's palm oil (Rudy *et al.* 2022 and Hamidi *et al.* 2022). Palm oil is largely consumed in food, beverages, cosmetics and pharmaceuticals. Nevertheless, it is a rich source of vitamins A and E, and low cost along with trans fatty acids response during heating and frying made it more valuable and preferable than other vegetable oils (Tripathi *et al.* 2022).

The health benefits and adverse impact both revolves around the type and quality of consumed edible oil. Crude vegetable oil is consisted of required nutri-

\*Corresponding author:

Innovation and Knowledge Center, Ashapura Minechem Limited  
Madhapar, Bhuj, Kutch, Gujarat, India 370020  
Email: indu.bajpai@ashapura.com

tion and unhealthy components as well. This undesired part from crude oil is partly separated by variety of processes to enhance oxidative stability and shelf life of oil (Chew *et al.* 2021). The removal of undesired phosphatides, trans fatty acid and gum while minimizing the oil loss during the process is the focus of degumming route (Carr *et al.* 1978). Although, degumming process passes through many steps and requires sophisticated facilities and raw materials as well.

The degumming of palm oil is crucial before processing for refining. In current era, a number of degumming routes are available and adopted by oil refining industries (Gharbi *et al.* 2022). Each and every degumming process influences the quality of processed oil and production cost as well. Some of the popular degumming routes include water degumming, enzymatic degumming, acid degumming, modified acid degumming and membrane filter degumming (Dijkstra *et al.* 2010 and Manjuka *et al.* 2007). Acid degumming is one of the oldest and convenient routes for removing phosphatides from the crude palm oil. The orthophosphoric acid, citric acid, nitric acid and some other edible grade acids are being used for the degumming purpose (Ohlsol *et al.* 1976). These acids were added in the barrel of oil to chelate with the gum to make complexes and settle down gum in the bottom of the barrel. The settling down phosphatides takes more than 8 hours. This degummed oil from top of the barrel is transferred for the further refining process.

The oil is treated with clay at ~110 °C to remove the colouring pigments. So, in the current work, authors have developed a novel product which will do degumming and bleaching at the same time in a single step. This product will save energy, time and resources to produce final refined palm oil which will benefit the oil refineries and ultimately the consumers in terms of price of product. In the consideration of degumming materials, clay is one of the best choices in terms of availability, cost,

nontoxicity and ease of processing. Combination of selected clay minerals was chosen in current work to produce degumming aid (named as ABA-degumming aid) for phosphorous removal. The chemical composition of clays received from different sources was critically analysed and used for performance check with different types of oil.

## MATERIALS AND METHODS

### Materials

Clay minerals were mined from the mines located in southern part of India. After processing and characterization of materials extracted from mines, silica, palygorskite and montmorillonite minerals were selected to prepare ABA-degumming aid. Crude palm oil was purchased from a renowned company located in Gujarat, India. All reagent grade chemicals like hydrochloric acid (37%, Merck), zinc oxide (99.99%, Finar), potassium hydroxide (99.9%, Merck), sulfuric acid (99%, Merck), sodium molybdate (99.9%, Merck), hydrazine sulphate (99.9%, Merck), cyclohexane and dihydrogen potassium phosphate (99.9%, Merck) were used in current study.

A number of apparatus like silicon oil bath with stirring at 250 rpm and heating up to temperature 110 °C, deodorization set up with vacuum pump, heating element and thermometer, hot plate with operating temperature up to 250 °C and muffle furnace with operating temperature up to 1100 °C were needed in the present work.

### Methods

ABA-degumming aid for palm oil was prepared using the existing facility in plant by raw material selection, sampling, sizing, surface modification, and blending process respectively. The adopted processing steps of clay-based ABA-degumming aid were described in Figure 1. The selected clay raw materials goes to the acid activation to improve the surface area and adsorptive performance then ground to create homogeneous powder with average size 12.7 µm. Subsequently, clay powder was blend-

ed with additive in ribbon blender and test samples from different sections of blender were taken out, mixed together and analysed. After performance evaluation of all test samples, product was discharged and stored in bags with liners.

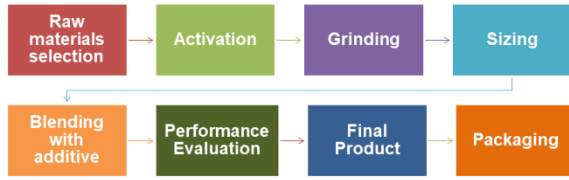


Figure 1 Process flow of preparation of ABA-degumming aid for edible oil.

### Bleachability Testing and Degumming of Oil

In order to conduct the bleachability testing of the oil, 40 g melted crude palm oil was poured in a glass cuvette and 1.4% (w/w) ABA-degumming aid was added and kept in the pre-heated oil bath at 105 °C for 30 min with stirring speed of 250 rpm. Subsequently, hot oil and ABA-degumming aid mixture was filtered with Whatman No-1 filter paper. The colour analysis (using red scale values) which corresponds to the transmittance of light through the oil was carried out at room temperature with 1-inch quartz cuvette for crude, bleached in Lovibond tintometer and bleachability was calculated as per given formula (Bayram *et al.* 2021).

$$\text{Bleachability (\%)} = \frac{\text{Red crude oil} - \text{Red treated oil}}{\text{Red crude oil}} \times 100$$

### Deodorization

After bleaching process, filtered oil was deodorized at 260 °C under 740-760 mmHg vacuum and without stirring for 45 minutes. The colour analysis (using red scale values) was carried out at room temperature with 1-inch quartz cuvette for crude and deodorized oil in Lovibond tintometer and bleachability (colour improvement after deodorization) was calculated by formula used above for bleachability calculation.

### Phosphorous Analysis

Phosphorous analysis of edible oil was performed by following ISO standard AOCS CA 12-55. In order to conduct the phosphorous analysis; crude palm oil was bleached with ABA-degumming aids and filtered. Afterwards 0.5 g ZnO was added to 10 g bleached oil and kept for charring in silica crucible on preheated hot plate at 250 °C for more than 24 hours. Furthermore, charred oil (black mass) was converted into ash at 600 °C temperature with 2 hours soaking time. Then hydrochloric acid was poured on the ash of the oil and immediately double distilled water was added. Samples were boiled on hot plate for 5 minutes then filtered and washed 5 times. As per the protocol, potassium hydroxide solution was added to turbid the solutions then precipitates were dissolved by addition of hydrochloric acid. All these solutions were prepared in 100 mL volumetric flask. Subsequently, 10 mL solution of this 100 mL solution was taken and hydrazine sulphate and sodium molybdate solutions were used for colour development in hot water bath. All test solutions along with fresh standard solutions (0, 2.5, 5, 10 and 20 ppm) of phosphorous were analysed in UV-Visible spectrophotometer at 650 nm wavelength.

### Free Fatty Acid and Acid Value Analysis

Free fatty acid (FFA) and acid value analysis (AV) of palm oil was performed as per ISO standard (ISO 660:2009 (EN)). Briefly, 10 g oil sample was taken in a flask and 2–3 drops of phenolphthalein indicator was added. Further 50 mL absolute ethanol was mixed to this solution at 65 °C temperature and allowed to cool down. Finally, this solution was titrated with 0.1 N KOH solution and Acid Value and FFA was calculated according to below formula:

$$\text{Acid Value} = \frac{\text{Concentration of KOH} \times \text{Volume of KOH} \times 56.1}{\text{Weight of oil sample}}$$

$$\text{FFA} = \frac{\text{Volume KOH} \times \text{Normality KOH} \times \text{Molecular weight FFA}}{56.1 \times 10 \times \text{Weight oil sample}}$$

### Carotenoids Content Analysis

Carotenoid analysis of oil was performed as per British standard BS684: section 2.20:1977. Briefly, in order to determine the carotenoid content in oil, 1 g oil sample was dissolved in a 100 mL flask with cyclohexane. The absorbance of such solution is measured at 446 nm in 10 mm path length quartz cuvette and carotenoid content is calculated according to below formula:

$$\text{Carotenoids (ppm)} = \frac{383 \times \text{Absorbance}}{\text{Path length} \times \text{Concentration of solution}}$$

### Deterioration of Bleaching Index

Deterioration of bleaching index (DOBI) analysis of oil was performed as per British standard BS-684:section 2.20:1977. Briefly, in order to determine the carotenoids content in oil, 1 g oil sample was dissolved in a 100 mL volumetric flask with cyclohexane. The absorbance of such solution is measured at 446 and 269 nm in 10 mm path length quartz cuvette and DOBI content is calculated according to below formula (Ifa *et al.* 2021).

$$\text{DOBI value} = \frac{\text{Absorbance at 446 nm}}{\text{Absorbance at 269 nm}}$$

In the current work, all experiments were repeated at least four times and always triplicate of sample were analysed.

### Characterizations of Clay Minerals

A chemical composition of the materials was studied by wavelength dispersive X-ray fluorescence technique (WD-XRF, Rigaku Primus III+) having as excitation source a miniaturized 30 KV X-ray tube. Mineralogy of clay samples was analysed by X-ray diffraction technique (XRD, Shimadzu Lab X6000). The 200-mesh pass (74-micron particle size) blend was scanned at 1°/min scan rate at 30 mA current and 40 KV voltage. For the quantification of phases, Siroquant software with Rietveld method was used. The image of gold coated ABA-degumming aid was scanned by table top scanning electron microscopy (SEM, JEOL 6510)

operated at 20 KV with Tungsten electron source. To measure the absorbance at 650, 446 and 299 nm UV-Visible Spectrophotometer (Specord 200 Plus Germany) was used. The red and yellow colour analysis of oil was conducted on Lovibond Colorimeter (PFX-i Series) and Tintometer (Lovibond Colorimeter model-F) at 1 and 10 inch path lengths. Particle size of degumming aid was analysed by Malvern particle size analyser (Mastersizer 2000) in wet system with sonication for 2 minutes at 12.5 Hz along with 1800-2200 rpm of hydro pump (Hydro 2000 MU) at room temperature. Brunauer-Emmett-Teller (BET, Quantachrome) was used to analyse surface area and pore volume.

## RESULTS AND DISCUSSION

The current study was started from the critical analysis of raw materials to formulate the most efficient product without compromising with the quality of obtained oil. The ABA-degumming aid was prepared by selection of specific clay minerals along with one additive in specific quantity. Figure 2 presents the mineralogical analysis of the ABA-degumming aid. Critical analysis of the diffraction pattern revealed the presence of montmorillonite and silica phases as a major constituent of the ABA-degumming aid. However, palygorskite, hematite, and calcite, phases were also detected in significant quantity. The characteristic peak of montmorillonite, palygorskite, hematite, quartz, and calcite was detected at 2θ value 6°, 8.5°, 35.6°, 26.6° and 29.22° respectively. The presence of these minerals is very crucial for the degumming/bleaching of edible oil. The clay used to produce ABA-degumming aid is consisted of group of minerals, presented in Table 1. The quantification analysis shows that 86% of total aid is constituted by montmorillonite, palygorskite and silica minerals and rest 14% is occupied by calcite and hematite minerals. The quantitative analysis of chemical composition of the ABA-degumming aid reveals the presence of silica, alumina,

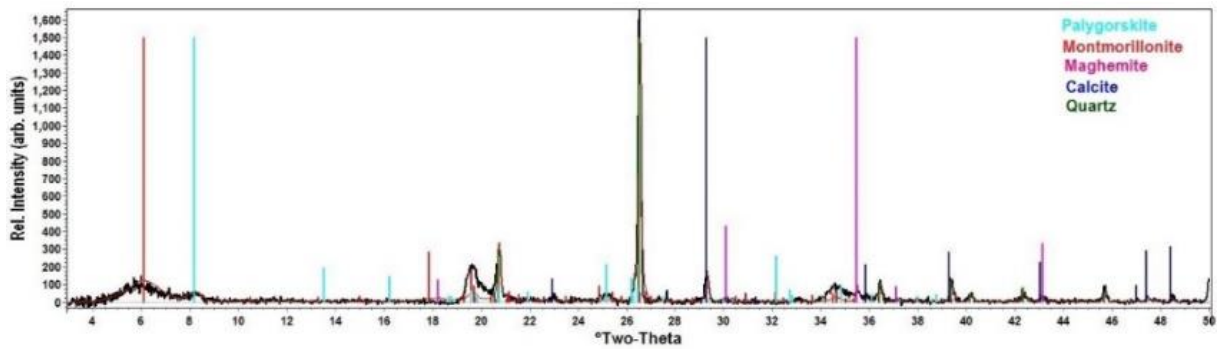


Figure 2 XRD pattern of the ABA-degumming aid.

iron oxide, calcium oxide, magnesium oxide and potassium oxide as significantly constituting chemicals of the ABA-degumming aid, which justified the mineralogical analysis well.

Table 1 Minerological composition of ABA-degumming aid

Phases	Quantification (%)
Palygorskite	16.0
Montmorillonite	49.0
Hematite	6.0
Silica	20.0
Calcite	9.0

Figure 3 shows the scanning electron microscopy image of the ABA-degumming aid. In order to ensure the uniform mixing of all constituents, five samples from different portion of the ribbon blender were drawn and images were captured by scanning electron microscope. The micrograph showed the uniform distribution of the raw materials and non-agglomeration. That confirms the physical blending of all raw materials is uniform. The uniform mixing of all constituents is a key factor to produce a quality product with consistent performance.

Certain physical parameters like bulk density, moisture and fineness directly impact the quality and cost of product. The ABA-degumming aid (phosphorous adsorbent) have 5.88% moisture which is very common in clay mineral based products. The fineness value plays important role in adsorption of colouring pigments present in oil and ABA-phosphorous adsorbent have 90% product of less than 90 µm size. Sieve

method was adopted for fineness analysis of the product. The loose bulk density of the product is 0.7 g/mL, which indicates that product, is in the free flow form and has high surface area (145 m<sup>2</sup>/g). Table 2 presents the surface area and pH value of natural attapulgite and ABA-degumming aid. The product has slightly acidic pH (5.8) value, which is the result of additive and surface treatment. This acidic pH favours the phosphorous removal from edible oil.

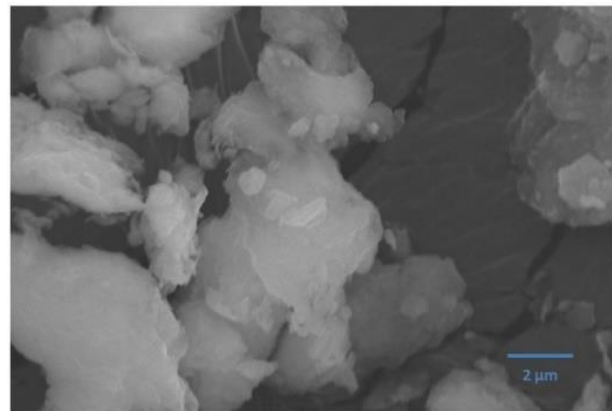


Figure 3 Scanning electron microscopy image of ABA-degumming aid.

Figure 4 shows the dose dependent response of ABA-degumming aid in terms of removal of phosphorous from the crude palm oil. The clay based degumming aid was analysed in a range of dose of aid and the higher value of phosphorous removed was set as a base to decide final dose. The dose of aid in edible oil should be in affordable range and it should not adversely alter the oil in terms of quality and nutrients. In the current study, 1.4% dose was found to reduce phosphorous up

to 96% and this dose was set as a benchmark for the further studies.

Figure 5 presents the response of ABA-degumming aid on crude palm and rice brane oil (RBO). The performance of ABA-degumming aid was found to be dependent on the type of oil. The dose of degumming aid is also very crucial and higher dose will also enhance the processing cost of the oil. The degumming aid dose 1.4% was observed to be effective in palm oil but 3% dose of same degumming aid was not so effective in RBO oil. So, on the basis of current results, different types of oil requires different types of degumming process, aid and dose also. Appreciably the 1.4% dose of ABA-degumming aid removed 96% phosphorous from the crude palm oil while 3% dose in RBO oil removed only 10% phosphorous. The degumming of RBO is critical and complex mechanism. RBO contains high free fatty Acid (FFA) and crude gums of RBO contains oil, phospholipids, and wax (Senger et al. 2014). Phosphorus content in the RBO oil (55 ppm) is almost 2.5

times than that of the palm oil (22 ppm) (Figure 5). This all makes it complicated and sometime it requires two stage degumming to get the desired phosphorus content.

Figure 6 presents the percentage bleachability of bleached and deodorized palm oil. The results depicts that 45% bleachability at bleaching stage was achieved with proposed degumming aid, which is good and support that ABA-degumming aid is as good as other degumming process. As far as the deodorization response is concerned, 95% colour improvement at deodorization stage was reasonably good but further scope of improvement still exists.

Table 3 presents some important parameters of edible oil to judge quality and suitability in products. The ABA-degumming aid showed remarkable reduction in certain harmful organic substances at bleaching stage. Deodorization is the final step of the oil refining process which removes all undesired free fatty acids (FFA) and carotenoids and reduce colour.

Table 2 Surface area and pH analysis of natural clay and ABA-degumming aid

Quality Parameter	Unit	Natural Clay (before processing)	ABA-Degumming Aid (after processing)
Specific BET Surface area	m <sup>2</sup> /g	90.0	145.0
pH	–	9.5	5.8

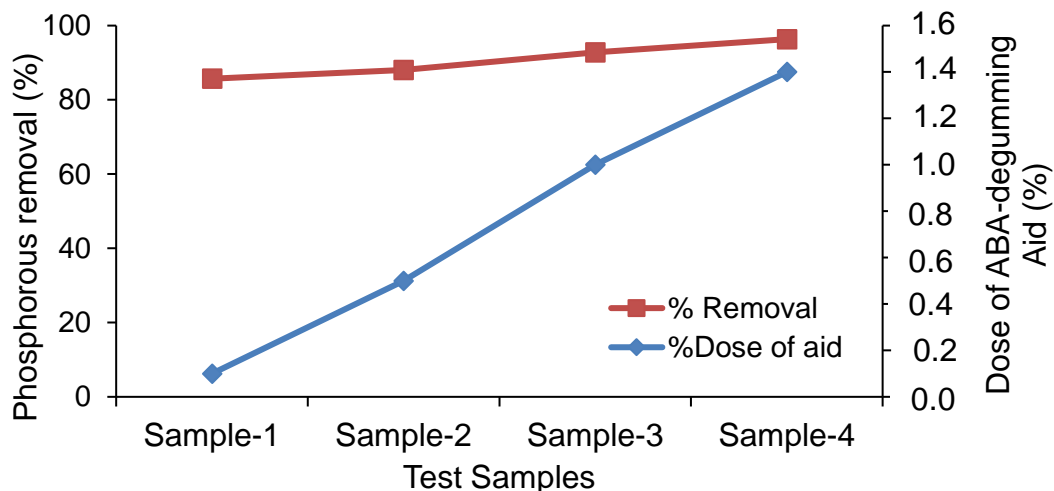


Figure 4 Response of degumming aid with respect to dose in crude palm oil.

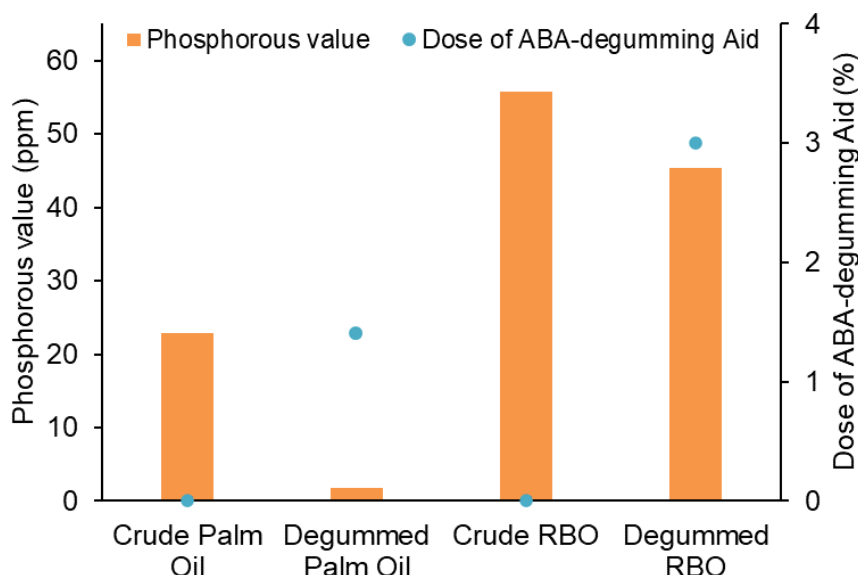


Figure 5 Response of degumming aid on crude palm and rice bran oil.

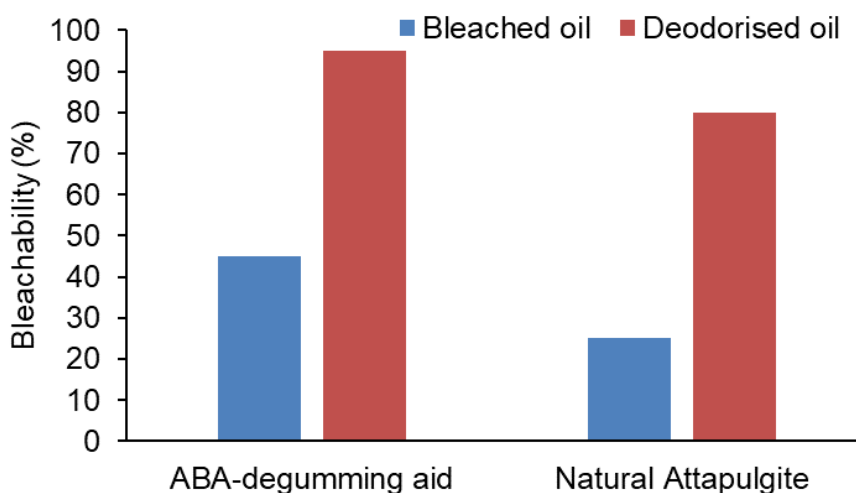


Figure 6 Bleachability at bleaching stage and deodorization stage of crude palm oil with ABA-degumming aid and natural attapulгите.

Table 3 Oil parameters of crude oil and refined with ABA-degumming aid

Oil	$\beta$ Carotenoids (mg/kg)	FFA (%)	Acid Value (mg KOH/g of substance)	DOBI Value	Colour (Red in 1-inch path length)
Crude Oil	400–600	4.0–6.0	8.0–12.0	1.8–2.2	20
Bleached Oil	40–60	4.0–6.0	8.0–12.0	–	12
Deodorized Oil	<5.0	<0.1	<0.2	–	1

The FFA, acid value, and carotenoids results shows the non-toxicity of ABA-degumming aid for the palm oil and colour values of the deodorized oil are remarkable which are much better than only attapulгите

bleached palm oil as presented in Figure 6. The removal of carotene is essential because  $\beta$ -carotene can react through cation, forming an alkyl radical (Krinsky *et al.* 2003 and Sarier *et al.* 1988). Figure 7 presents

the stability results of the ABA-degumming aid. In the current work, ABA-degumming aid was stored in a bag to conduct stability response up to six months. Each time, test sample was drawn from the bag and bag was again tightly sealed. Every time

samples were tested for phosphorous removal and bleachability. The samples were found to show consistent performance up to six months. Each time 18 crude oil was found to comprise different phosphorous value.

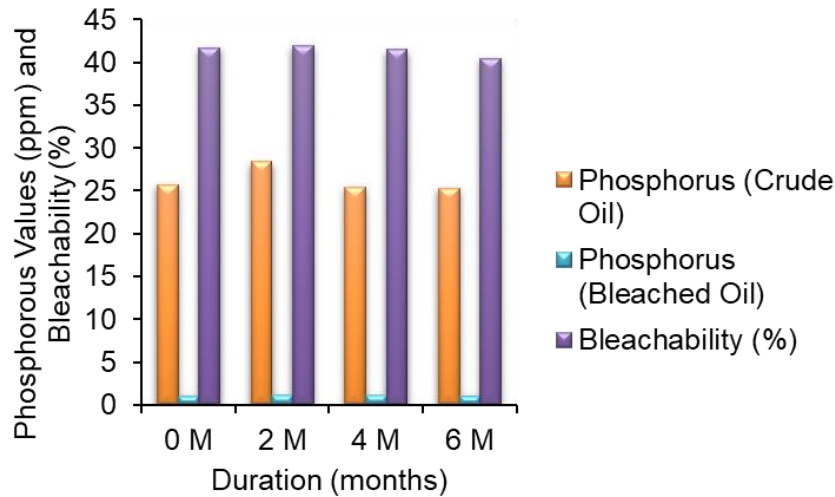


Figure 7 Stability analysis of ABA-degumming aid up to 6 months from manufacturing day (0 months), M stands for month.

In general degumming does not take place during bleaching process however it requires separate step like acid treatment, enzymatic treatment, water degumming, membrane filter degumming and many more ways. Degumming along with bleaching step is the main advantage of the ABA-degumming aid. This two in one utilization reduces time to refine oil, additional infrastructure, facilities, materials and waste management as well. The compilation of all these major steps directly impacts the selling price of edible palm oil which benefits the consumers. Most of the commercial oil refining companies use orthophosphoric acid (0.02–0.5% at 90–110 °C for 10–30 min) for the removal of phosphorous from the crude palm oil (Ebuna *et al.* 2007, Nur *et al.* 2021 and Irvan *et al.* 2020). This step is sophisticated and expensive as well, despite of low required dose. A number of products have been reported to lighten the colour of palm oil in bleaching process (Silva *et al.* 1992, Zschau *et al.* 2001, and Boki *et al.* 1992). Attapulgite is the main clay which is commercially used for the bleaching of

palm oil and surface modification by thermal/acid activation or carbon modification creates more pores on the surface of clay which helps in removing by trapping and adsorbing the colouring pigments from the palm oil (Gaugyan *et al.* 2018). As per the available literature 1 to 3% dose of attapulgite was reported to be used for bleaching of palm oil. But for degumming process acid is used. In contrast, in the present work only 1.4% dose bleached the crude palm oil and degummed as well. Comparison between major steps of oil refining process with phosphoric acid and ABA-degumming aid were presented in Figure 8.

The presence of phosphorous directly reduces the oxidative stability of the refined oil. ABA-degumming aid removed 96% phosphorous from the crude palm oil. The mechanism behind the phosphorous removal by ABA-degumming aid is the additive used along with modified clay make metal complex with the impurities (phosphorous, iron, heavy metals) present in the crude oil and as after the bleaching process, oil is filtered then



phosphorous presented in gum was attached on aid, is separated out from the oil. Figure 9 presented the representative model of the removal of phosphorous (gums), colouring pigments and other impurities from the crude palm oil by using developed ABA-degumming aid. The opti-

mum combination of clays and additive, with intercalation and positive charge on the surface and structure as well, are supposed to be directly responsible for removing the gums, colouring pigments and other types of impurities associated with the oil.

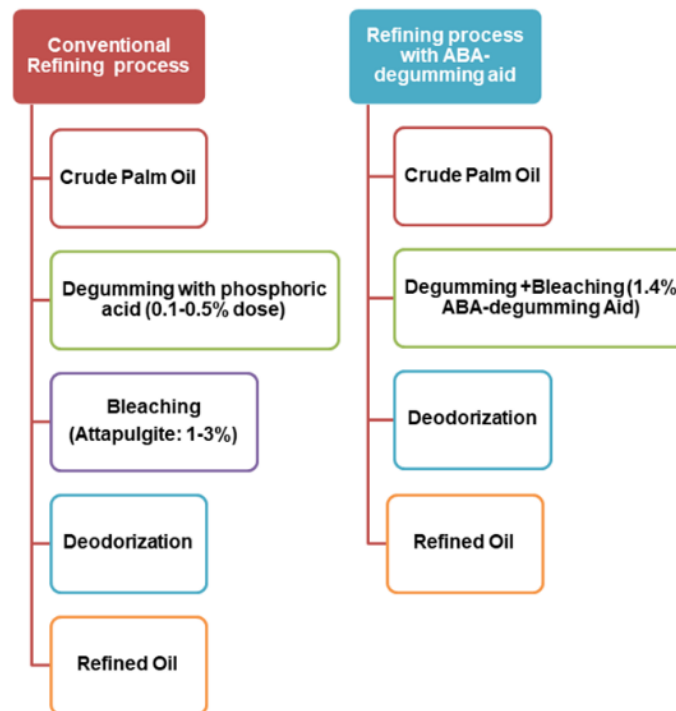


Figure 8 Comparative representation between oil refining process with phosphoric acid and ABA-degumming aid.

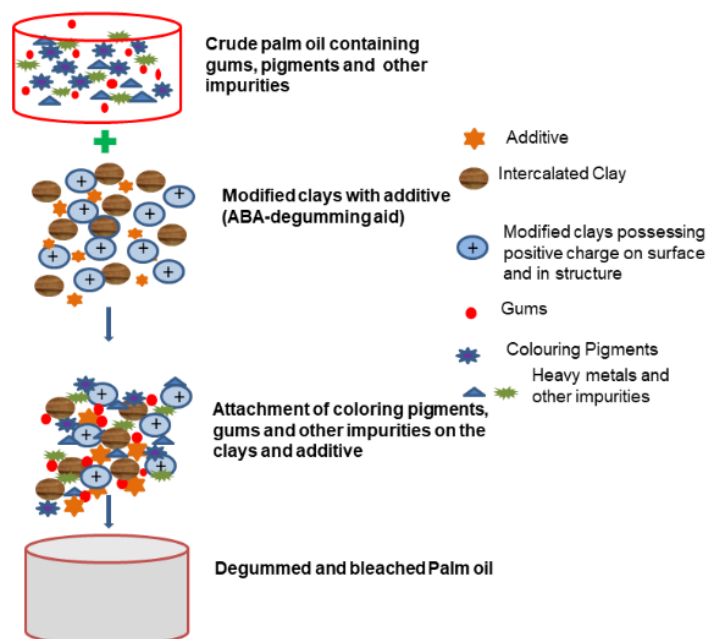


Figure 9 Representative model of bleaching along with degumming of palm oil by using ABA-degumming aid.

Clay minerals are abundant on earth and has been used as a bleaching clay adsorbent for the refining of edible oil like palm oil, soybean oil, sunflower oil etc. (Lin *et al.* 2007). Clays like palygorskite and montmorillonite possess surface area which allows the researcher to use it as adsorbent. This surface area is further enhanced by doing the acid activation which in results helps to promote the adsorption of carotenoids and phosphorous from palm oil (Wie *et al.* 2004). The acid activated clays gives good bleaching performance but fails to give the good phosphorus removal at the bleaching stage (Silva *et al.* 2013).

To overcome these issues, authors have modified the bleaching clay with additive, which helps to removed up to 96% phosphorous along with the bleachability at bleaching stage (105 °C). The additive is intercalated with the high surface area clay (Table 2). The additive act as chelating agent which helps to remove the phosphorous and high surface area clay act as a bleaching clay which helps to remove the carotenoids. The pH of ABA-degumming aid is lightly acid which creates the favourable condition for phosphorous and carotenoids removal.

The nonhydratable phosphatides are present in the form of calcium or magnesium salt of phosphatides (Vintila 2009). The adsorbent ABA-degumming aid forms a complex with calcium and magnesium. Calcium and magnesium salts of phosphatides get adsorbed on the surface of ABA-degumming aid. The high surface area of ABA-degumming aid adsorbed the carotenoids and gives the bleaching performance. Both the adsorbed contaminants (phosphatide complex and carotenoids) are removed at the filtration stage.

Conventional degumming process of CPO takes around 10–12 hours to settle the gums in the bottom after the reaction in hot condition while bleaching requires only 0.5 hour. Additionally, settled gum carry 10% oil (with respect to weight of gum) which is considered as processing loss.

Therefore, conducting degumming and bleaching in a single step by using ABA-degumming aid will reduce oil loss and processing time. Moreover, additional infrastructure for degumming will not be required. In the conventional process, phosphoric acid is used to remove the non-hydratable phosphatide in the dose of 0.1–0.5% (Lin *et al.* 2007). The average price of phosphoric acid varies in range of 75–95 Indian Rupees (INR), whereas the cost of ABA-degumming aid is 30–40% of the phosphoric acid. In the conventional process (Figure 8) degumming with phosphoric acid and Bleaching is done in two separate stages, whereas ABA-degumming aid completes both the process in a single stage. The proposed process by authors saved the asset cost of degumming tank, processing cost and energy cost of agitation, storage, and addition of phosphoric acid. Save the occupied space of degumming tank. The conventional degumming requires min 10–12 hours, whereas in the proposed process by authors the degumming is completed with bleaching only, so does not ask for separate time. Overall, eliminating separate degumming step will help to maintain quality, enhance yield, save time and reduce cost of production.

## CONCLUSION

The ABA-degumming aid for the removal of phosphorous from the crude palm oil was prepared successfully. This product is economically suitable for refining of crude palm oil. The ABA-degumming aid suppressed separate degumming step while degumming and bleaching was done simultaneously in a single step. The response of ABA-degumming aid was not much prominent in RBO oil, it was only useful for the palm oil. The bleachability of bleached palm oil and deodorized palm oil was around 45% and 95% respectively, which is also an advantage with the proposed degumming aid. On the basis of conducted experiments and analysis, ABA-degumming aid is a good quality product

which is useful and economic for the palm oil refining industries.

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