



## PERFORMANCE OF PALM SHELL ACTIVATED CARBON AS AN ALTERNATIVE ADSORBENT FOR RECLAMATION OF USED TRANSFORMER OIL

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### ABSTRACT

The objective of this study is to investigate the effectiveness of three types of adsorbents, namely Fuller's earth, bentonite and palm shell activated carbon, on the reclamation of used transformer oil samples. Fuller's earth is an industrial bleaching earth that is commonly used as an adsorbent in the reclamation process, whereas bentonite and palm shell activated carbon are alternative adsorbents that are relatively new in the field. In this study, the reclamation process is carried out on the used transformer oil samples five times, and the effectiveness of the adsorbents after the first, third and fifth reclamation cycle is examined. The relative content of dissolved decay products (DDP) for each sample before and after the reclamation process is determined using UV-Vis spectroscopy. In addition, the breakdown voltage test is performed to measure the dielectric strength of each sample under an applied electric field in accordance with the ASTM D6802 and MS IEC 60156:2012 standards. The reclamation process conducted in this study complies with the BS EN 60422:2013 and IEEE Std 637-1985 (R2007) standards. The results show that both bentonite and palm shell activated carbon adsorbents reduce the relative content of DDP by twice the value achieved by Fuller's earth, particularly during the first and third reclamation cycles, while simultaneously enhancing the dielectric strength of the reclaimed oil samples. The results also show that the use of palm shell activated carbon gives the highest percentage of enhancement in the breakdown voltage test, with a value of 57.14% after the fifth reclamation cycle. Based on the results of this study, it can be concluded that palm shell activated carbon is a promising alternative adsorbent for reclamation of used transformer oils, which diversifies the uses of palm oil products in Malaysia.

**Keywords:** transformer oil, reclamation, adsorbent, UV-Vis, breakdown voltage, oxidation.

### INTRODUCTION

It is known that transformer oils have been primarily used as electrical insulations and cooling media (by enhancing heat transfer). Transformer oils are also used as informative tools to diagnose the conditions of transformers [1], [2]. These oils can be used to extend the lifetime of transformers by 25 to 30 years [3]. However, transformers are also subjected to electrical, thermal and chemical stresses in during their lifetime [4], [5]. The presence of oxygen and moisture leads to degradation of the transformer oil, making it acidic. Consequently, the dielectric strength of the insulation paper is reduced.

The by-products of the oxidation process can be either soluble or insoluble. The soluble by-products can be measured using UV-Vis spectrometry, Fourier Transform Infrared Spectrometry (FTIR), total acid number (TAN), dissipation factor ( $\tan \delta$ ) and interfacial tension (IFT) [6], whereas the insoluble by-products are quantified based on turbidity and particle counts, as well as judged qualitatively based on the colour and appearance of the transformer oil [7]. The soluble by-products are simply termed as dissolved decay products (DDP), which are produced from the build-up of peroxides, aldehydes, ketones and organic acids [5]. The DDP will partially adsorb in the insulation paper and pressboard, which degrades their dielectric strength and reduces reliability of the transformer [8].

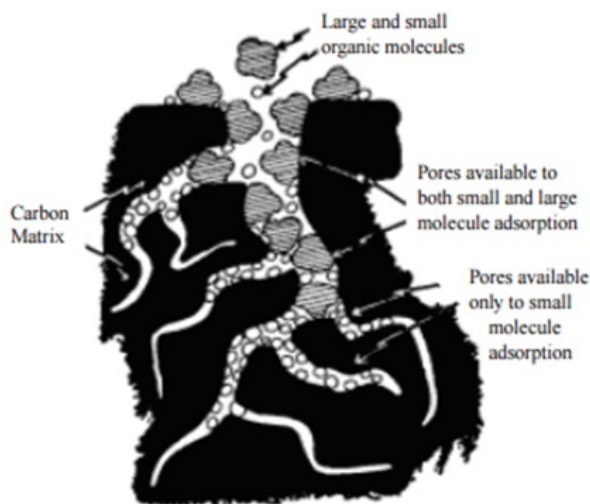
Reclamation of used transformer oils has been developed since the early 1980s [1], [9]. The reclamation process basically regenerates the essential properties of the in-service transformer oil, such that the quality of the reclaimed oil is equivalent to that of new transformer oil. Reclamation is an attractive recycling technique because it is both economical and environmental-friendly [10]. Two standards have been established that serve as guidelines for the operation and maintenance of transformers, namely BS EN 60422: 2013 and IEEE Std 637-1985 (R2007) [11], [12]. A comprehensive description of the reclamation process of transformer oil and the desired physical, electrical and chemical properties is given in IEEE Std 637-1985. The reclamation process involves adsorption, filtration, dehydration and degasification. According to the BS EN 60422:2013 standard [12], there are two types of reclamation process: (1) reclamation by percolation and (2) reclamation by contact. Each of these processes is described briefly as follows.

- 1) Reclamation by percolation: This is an on-site process which involves filtering, adsorption and reconditioning.
- 2) Reclaiming by contact: This is a laboratory technique which involves stirring the contaminated oil with an adsorbent, specifically Fuller's earth (FE), in a suitable container.



Adsorbent is one of the essential elements in the reclamation of used transformer oils since it is used to impede DDP, especially soluble by-products [3], [10], [13], [14]. Filtration, dehydration and degasification are focused on eliminating insoluble by-products such as moisture content, solid particles and dissolved fault gases [11], [12]. In recent years, researchers have introduced new adsorbents that are comparable with existing industrial adsorbents (FE). These alternative adsorbents include activated alumina, bentonite, membrane, activated Fuller's earth and activated carbon [3], [10], [13], [15].

In this study, palm shell activated carbon (PSAC) is selected as the adsorbent for reclamation of used transformer oil. This product has been used in various industries which include water treatment, gas purification, air purification and spill clean-up [16], [17]. Figure-1 shows the capability of activated carbon pores and structures in adsorbing certain materials. Activated carbon based on different materials have also been used as adsorbents for recycling used lubricant oils [18], [19]. Owing to the capabilities of PSAC, the objective of this study is to investigate the effectiveness of PSAC in reclaiming used transformer oil. This adsorbent is an attractive alternative due to the following reasons: (1) It is economical; (2) It is easily attainable since it is manufactured locally in Malaysia; (3) It can reduce the number of reclamation cycles (which speeds up the reclamation process) since the adsorbent can remove higher relative content of DDP during the first reclamation cycle; and (4) It can be reactivated after use.



**Figure-1.** Activated carbon particle pores and structures [16].

This study is centred on three types of adsorbents (FE, bentonite and PSAC) in order to determine the best adsorbent for the reclamation process. FE and bentonite are essentially natural adsorbent clays which enable the non-polar components of the oil to pass through without retention, and retains only the polar contaminants or degradation compounds dissolved in the oil [12]. These

adsorbent clays consist of silicate anions ( $\text{Si}_2\text{O}_5$ )<sub>n</sub> condensed with octahedral layers of  $\text{X}(\text{OH})$ . It shall be noted here that 'X' may be magnesium, aluminium, etc. [11], [12]. PSAC is a relatively new adsorbent based on palm shells. A new adsorbent based on palm shells is also introduced for this purpose, which adds value to local palm oil products in Malaysia. UV-Vis spectrometry and breakdown voltage test were used to evaluate the effectiveness of each adsorbent on reclamation of used transformer oil.

## METHODOLOGY

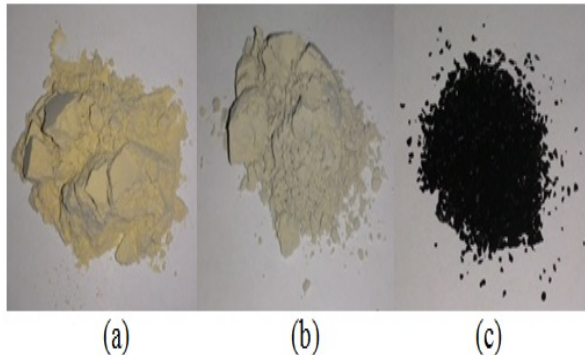
### a) Sample preparation and reclamation process

The used transformer mineral-based oil used for all samples in this study was sourced from TNB Research Malaysia, a subsidiary company of Tenaga Nasional Berhad, which is the main power provider in Malaysia. The FE and bentonite adsorbents used for the reclamation process were purchased from Sigma-Aldrich, whereas the PSAC adsorbent was supplied by Green Scientific Enterprise, Malaysia. These adsorbents are summarized in Table-1, and their corresponding photographs are shown in Figure-2. The procedure for sample preparation is described as follows. Firstly, 500 ml of used transformer oil was mixed with 5 g of FE, bentonite and PSAC, respectively. A hot plate magnetic stirrer was used to homogenize the concentrations at a temperature of 80°C and stirring speed of 750 rpm for a period of 1 hour [3]. Secondly, each oil sample was filtered using the gravity filtration technique. Whatman Filter 6 (3 microns) quantitative paper was used for this purpose, whereby the filter paper was first folded to form a cone and then placed into a filter funnel. The clean oil sample (filtrate) was collected in an Erlenmeyer flask. Photographs of the first and second steps of the reclamation process are shown in Figure-3. Finally, the oil samples were individually degassed and dried at a pressure less than 1 kPa for two days at 80°C. An additional day was needed to cool the oil samples to ambient temperature under vacuum conditions [20]. The procedure was repeated for five cycles due to time constraints in completing the gravity filtration process. Tests were also conducted on a new transformer oil sample in order to provide baseline data for comparison.

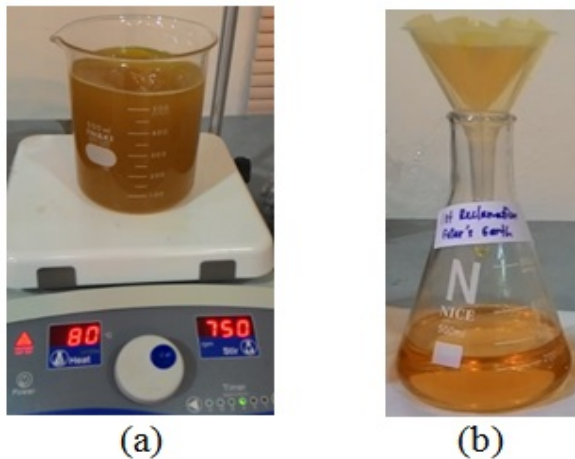
**Table-1.** List of adsorbents used in this study.

Adsorbent	Base material	Abbreviation
Fuller's earth	Bleaching clay	FE
Bentonite	Bleaching clay	N/A*
Palm shell activated carbon	Activated carbon (charcoal)	PSAC

\*Note: N/A denotes 'Not applicable'.



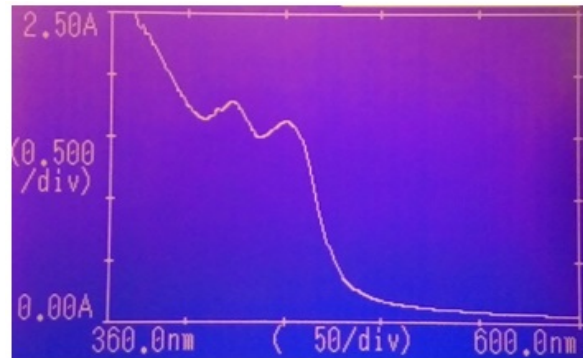
**Figure-2.** Photographs of the adsorbents used in the reclamation of used transformer oils: (a) Fuller's earth, (b) bentonite and (c) palm shell activated carbon.



**Figure-3.** Photographs of the first and second steps involved in the reclamation of used transformer oils: (a) mixing of oil with adsorbent using hot plate magnetic stirrer and (b) gravity filtration.

#### b) UV-Vis spectra measurements

Once the reclamation process was complete, the ultraviolet/visible (UV-Vis) spectrum of each oil sample was recorded using Shimadzu UV Mini 1240 UV-Vis spectrophotometer having a wavelength range of 190–1100 nm and equipped with quartz cells having a path length of 10 mm. The optical absorption of the samples was recorded over a wavelength range of 360–600 nm in accordance with the ASTM D6802 (2010) standard in order to determine the relative content of DDP for each mineral insulating oil [13], [21]. Figure-4 shows an example of a UV-Vis absorption spectrum recorded using the Shimadzu UV Mini 1240 instrument. The relative content of DDP of the oil sample is determined by integrating the area under this curve.



**Figure-4.** Example of UV-Vis absorption spectrum of a used transformer oil sample.

#### c) Breakdown voltage test

Megger OTS60PB portable oil test set was used to measure the AC breakdown voltages in accordance with the MS IEC 60156:2012 standard [22] using brass spherical electrodes. The electrode gap was set to 2.5 mm, and the rate of voltage was 2.0kV/s. A total of six breakdowns were performed for each oil sample and the mean breakdown voltage was calculated. The temperature of the test was maintained at  $30 \pm 5^\circ\text{C}$ . The oil samples were given a delay time of 2 minutes in between tests to enable them to cool to the desired temperature of  $30^\circ\text{C}$ .

### RESULTS AND DISCUSSION

#### a) Physical change of oil samples

When the transformer oil is used in service, it is exposed to high levels of oxidation, depending on internal and external factors. The colour of the oil is usually used to indicate the presence of oxidation and the degree of deterioration of the service-aged transformer oil [20]. Figure-5 shows the colour of the four oil samples before and after the reclamation process. It can be observed that the colour of the oil prior to reclamation (sample A) is brown due to oxidation. It is apparent that the colour of the oils becomes clear yellow after the reclamation process since some of the by-products of oxidation have been removed, as indicated by samples B, C and D.



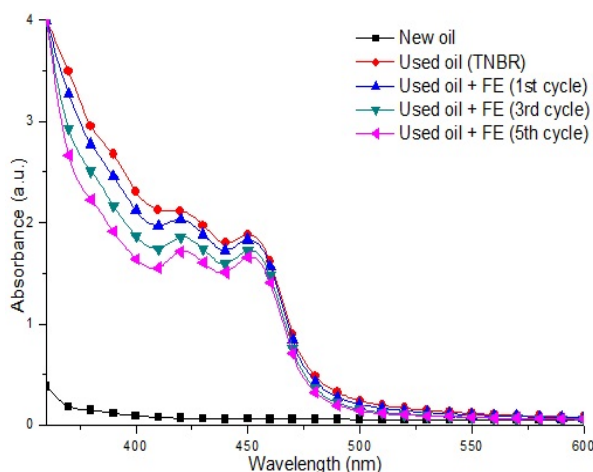
**Figure-5.** Colour of the oil samples, indicating the effectiveness of the reclamation process: (A) oil before reclamation, (B) oil after 5th reclamation cycle with Fuller's earth, (C) oil after 5th reclamation cycle with bentonite, and (D) oil after 5th reclamation cycle with PSAC.



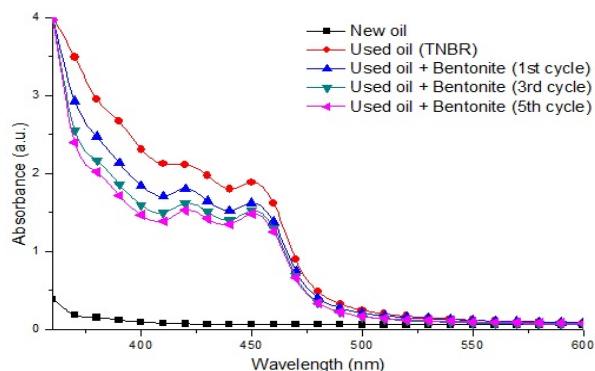


## b) UV-Vis analysis

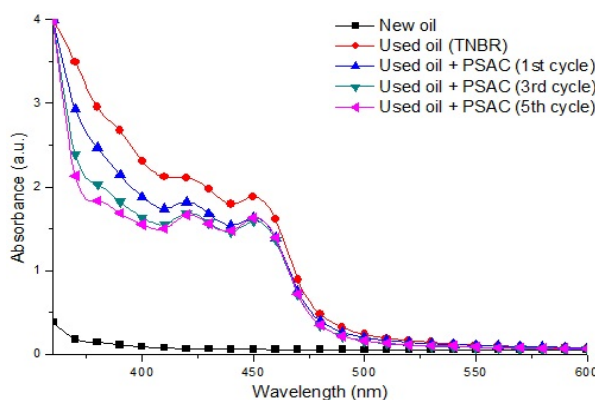
The absorbance curves of the used transformer oil samples reclaimed with FE, bentonite and PSAC are shown in Figure-6, Figure-7 and Figure-8, respectively. Wavelength of 400 nm is chosen as a reference for describing the results. It can be seen from these figures that the absorbance of the oil sample at a wavelength of 400 nm is 2.3068 a.u. prior to reclamation. In general, the results indicate that the by-products of oxidation decrease after the reclamation process, as evidenced from the absorbance curves. It is found that the absorbance of the oil sample at 400 nm decreases to 2.1177 a.u. (8.2%), 1.8701 a.u. (19%) and 1.8011 a.u. (22%) after the first, third and fifth reclamation cycle with FE, as shown in Figure-6. Likewise, the absorbance of the oil sample at 400 nm decreases to 1.8385 a.u. (20%), 1.5914 a.u. (31%) and 1.4645 a.u. (37%) after the first, third and fifth reclamation cycle with bentonite, as shown in Figure-7. A similar trend is observed when PSAC is used in the reclamation process, whereby the absorbance of the oil sample at 400 nm decreases to 1.8807 a.u. (18.5%), 1.6368 a.u. (30%) and 1.5529 a.u. (37%) after the first, third and fifth reclamation cycle, as shown in Figure-8. The absorbance curves at 400 nm of the oil samples reclaimed with FE, bentonite and PSAC after the fifth reclamation cycle are shown in Figure-9. It can be seen that the absorbance at 400 nm decreases from 2.3068 a.u. to 1.8011 a.u. (22%), 1.4645 a.u. (37%) and 1.5529 a.u. (33%) for used oil reclaimed with Fuller's earth, bentonite and PSAC, respectively. In general, the results indicate that bentonite gives the highest reduction of oxidation by-products compared to FE and PSAC at a wavelength of 400 nm.



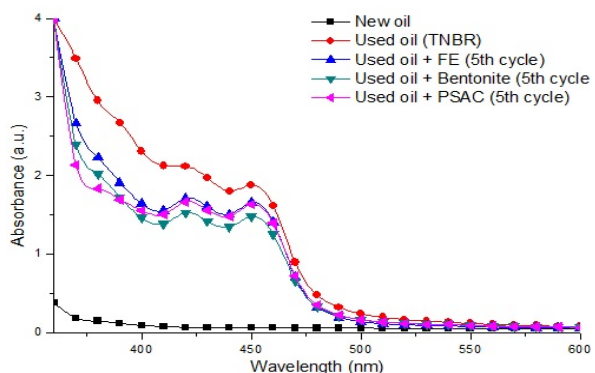
**Figure-6.** UV-Vis absorption spectra of used oil samples reclaimed with Fuller's earth.



**Figure-7.** UV-Vis absorption spectra of used oil samples reclaimed with bentonite.



**Figure-8.** UV-Vis absorption spectra of used oil samples reclaimed with palm shell activated carbon.

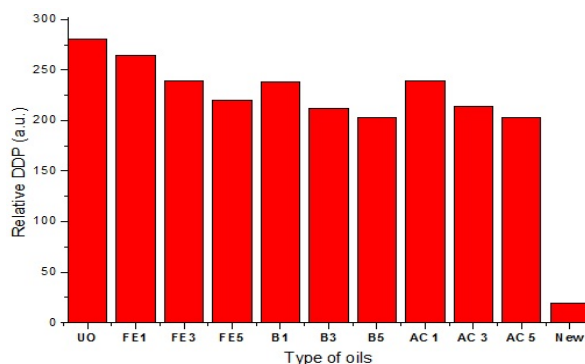


**Figure-9.** UV-Vis absorption spectra of used oil samples reclaimed with Fuller's earth, bentonite and palm shell activated carbon after the 5th reclamation cycle.

Figure-10 shows the relative content of DDP removed during the first, third and fifth reclamation cycles for all oil samples, which is determined from the area under the UV-Vis absorption curves within the following limits: 0–4 a.u. (absorbance) and 360–600 nm (wavelength). Table-2 shows the relative content of DDP and the percentage reduction of DDP for each reclaimed oil sample relative to the used transformer oil. It can be



observed from Table-2 that a large number of reclamation cycles are required to reclaim used transformer oils in order to remove the by-products of oxidation and yield oils with a quality that is comparable to the quality of new transformer oil. Based on the results, it is evident that the use of bentonite and PSAC adsorbents removes higher relative content of DDP compared to FE. Based on the area under the UV-Vis absorption spectrum, the relative content of DDP is 18.908 for new oil, which conforms to the value prescribed in the ASTM D6802 (2010) standard. According to this standard, the area under the UV-Vis absorbance spectrum is typically less than 25 Abs.  $\times$  nm for new oils [16]. In contrast, the relative content of DDP is rather high for the used transformer oil prior to reclamation, with a value of 280.223, which indicates that the oil has undergone oxidation during service. In general, the lower the relative content of DDP, the better the condition of the transformer oil. It can be seen from the results that bentonite (B1) and PSAC (PSAC1) reduces the relative content of DDP by 15 and 14.5%, respectively, during the first reclamation cycle. In contrast, the FE adsorbent (FE1) only decreases the relative DDP by 5.8%. A similar trend is also observed during the third reclamation cycle, whereby the bentonite (B3) and PSAC (PSAC3) removes the relative content of DDP by 24.3 and 23.6%, respectively, whereas the relative content of DDP removed by FE (FE3) is only 14.8%. The performance of both bentonite and PSAC adsorbents (B5 and PSAC5) is equal during the fifth reclamation cycle, whereby the relative content of DDP removed from the used transformer oil samples is 27.6%. In contrast, Fuller's earth reduces the relative content of DDP by 21% in the fifth reclamation cycle. Based on these results, it can be deduced that both bentonite and PSAC adsorbents are capable of reducing the relative content of DDP by roughly twice the value achieved for FE, which is evident during the first and third reclamation cycles. However, it can be observed that even though the bentonite and PSAC adsorbents outperform FE in the fifth regeneration cycle, the difference in the reduction in relative content of DDP between these three adsorbents is not as marked compared to the first and third regeneration cycles.



**Figure-10.** Relative content of DDP removed from the used oil samples during the first, third and fifth reclamation cycles.

**Table-2.** Relative content of DDP of used oil samples determined from the area under each UV-Vis absorption spectrum.

Oil sample	Relative content of DDP (area under the UV-Vis absorption spectrum)	Percentage of reduction (%)
New oil (New)	18.908	–
Used oil before reclamation (UO)	280.223	Baseline
Oil after 1 <sup>st</sup> reclamation cycle + Fuller's earth (FE1)	263.960	5.8
Oil after 3 <sup>rd</sup> reclamation cycle + Fuller's earth (FE3)	238.736	14.8
Oil after 5 <sup>th</sup> reclamation cycle + Fuller's earth (FE5)	220.135	21
Oil after 1 <sup>st</sup> reclamation cycle + Bentonite (B1)	237.888	15.1
Oil after 3 <sup>rd</sup> reclamation cycle + Bentonite (B3)	212.230	24.3
Oil after 5 <sup>th</sup> reclamation cycle + Bentonite (B5)	202.969	27.6
Oil after 1 <sup>st</sup> reclamation cycle + Palm shell activated carbon (PSAC 1)	239.451	14.5
Oil after 3 <sup>rd</sup> reclamation cycle + Palm shell activated carbon (PSAC 3)	214.007	23.6
Oil after 5 <sup>th</sup> reclamation cycle + Palm shell activated carbon (PSAC 5)	202.854	27.6

### c) Breakdown voltage test results

Table-3 shows the results of the breakdown voltage test for each oil sample before and after reclamation. The increase in the percentage of enhancement shows the effectiveness of the reclaimed oils compared to the used transformer oil in the electric field. The breakdown voltage (BdV) of the transformer oil is primarily dependent on four factors: moisture content, formation of bubbles, suspended solid particles and acidity of the oil [23], [24]. It can be observed from Table-3 that the BdV values of the reclaimed oil samples are generally higher compared to the value for used transformer oil before reclamation, regardless of the adsorbent used (FE, bentonite or PSAC). The percentage of enhancement varies for each reclaimed oil sample, depending on the removal of the relative content of DDP, moisture and solid particles during the reclamation process. Nevertheless, the percentage of enhancement is highest for PSAC compared to bentonite and FE, with a value of 43.75, 52.63 and 57.14%, respectively. Hence, the difference in the



percentage of enhancement between PSAC and bentonite is 4.51%, whereas this difference is even more pronounced between PSAC and Fuller's earth, with a value of 13.39%.

**Table-3.** Breakdown voltage test results.

Oil sample	Breakdown Tests (kV)						Mean (kV)	Percentage of enhancement (%)
	1	2	3	4	5	6		
Used oil before reclamation	9	9	10	10	8	9	9	Baseline
Oil after 5 <sup>th</sup> reclamation cycle + Fuller's earth	20	14	18	13	14	17	16	43.75
Oil after 5 <sup>th</sup> reclamation cycle + Bentonite	25	14	16	18	21	18	19	52.63
Oil after 5 <sup>th</sup> reclamation cycle + Palm shell activated carbon	21	20	23	16	28	15	21	57.14

## CONCLUSIONS

In this study, a series of experiments have been carried out to reclaim used transformer oils, beginning with the laboratory set-up of the reclamation process, followed by the selection of adsorbents, mixing of used oil samples with the respective adsorbents, and filtration by means of gravity filtration. The effectiveness of three types of adsorbents (FE, bentonite and PSAC) on the quality of the reclaimed transformer oils has been determined using UV-Vis spectroscopy and breakdown voltage test. Based on the results, it can be concluded that the bentonite and PSAC adsorbents outperform FE in removing the relative content of DDP, particularly during the first and third reclamation cycles. In addition, the percentage of enhancement in breakdown voltage is highest for the PSAC adsorbent. Hence, it is concluded that PSAC has great potential for use as an adsorbent in the reclamation of service-aged transformer oils and this helps diversify the uses of palm oil in Malaysia

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