

## Effectiveness of Olive-Waste Ash as an Adsorbent Material for the Regeneration of Fried Sunflower Oil

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### ABSTRACT

The present work explored the use of olive-waste ash, a byproduct of the manufacture process of olive oil in mills as a natural adsorbent for improving the quality of fried sunflower oil compared with synthetic adsorbent material (Magnesol XL) were used to absorb the oxidation products of fried sunflower oil. The metal content (Si, Mg, Ca, Fe, Al, Mn, and Cu) of the adsorbent materials were determined. The physico-chemical properties (color, viscosity free fatty acids, peroxide value, induction period, polar content and polymer content) of fresh, fried and fried-treated sunflower oil were determined. The frying process was carried out at 180°C±5°C for 24h, 4h heating cycle per/day for six consecutive days. The fried sunflower oil was treated with synthetic and natural adsorbent materials at 105°C for 15 min. The results indicate that Magnesol XL and olive waste ash contained Si<sup>+</sup> Mg<sup>+</sup> Mn and Si<sup>+</sup> Mg<sup>+</sup> Ca as the basic metals, respectively. Frying of sunflower oil led to significant increase in physico-chemical properties. Treatments of fried sunflower oil with the aforementioned substances greatly improved the quality of fried oil. The results of this study were shown that natural adsorbent (olive-waste ash) can improve its adsorption capacity significantly. In this respect, utilization of olive waste-ash for frying oil recovery applications can be an industrially is a very cheap and easily found material.

**Key words:** Adsorption, frying process, olive waste ash, regeneration, sunflower oil.

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### Introduction

Despite many negative aspects, food frying is still one of the most preferred food preparation techniques worldwide (Rossell, 2001 & Abadedunye and Przybylski, 2009). Frying is immersing and cooking foods in hot oil which serves as the heat and mass transfer medium. During the process, the presence of high temperature, oxygen, moisture and minerals leaching from the food cause a variety of reactions (oil hydrolysis and oxidation, dimmer and polymer formation, Maillard reactions etc.) to deteriorate the oil, and reduce the frying oil quality and healthiness properties of the fried products (Dulger & Yilmaz, 2013). The worldwide production of edible vegetable oil is about 60 million tons a year (United states Department of Agriculture, 2000), the majority being used for frying foods and a significant quantity of the used frying oils being discarded. Due to the increase of oil price of the developed countries to reduce biofuels from vegetable oils, there is an urgent need to extend the useful life of the used oils. therefore, some actions like regular cleaning and maintained of equipment, selection of good quality frying oil, adjustment of proper frying conditions, addition of some natural antioxidant extracts into the oil (Basuny, 2004; and Basuny *et al.*, 2013), and use of some adsorbents for removing soluble degraded compounds from the frying oil have been practice (Farg & Basuny 2002; and Farag *et al.*, 2009). These methods can regenerate the frying oil quality based on the removal of the harmful compounds formed during frying; therefore, treated oils can be used again in deep-fat frying process. Concern Farag and Basuny (2009) used inorganic (normal and modified silica gel) and organic (normal and modified cellulose powder) substances as regeneration agents for fried sunflower oil. These results demonstrated that these adsorbent were effective in improving the overall quality of used sunflower oil. Many reports have been appeared on the production of low cost adsorbents using cheaper and readily available material for example, Basuny and Arafat (2014) used activated carbon from date pits for removing peroxides, polymer and enhancing the stability of the frying oil. In this regard, residues from agriculture and agro-industries are the non-product outputs from the growing and processing of raw agricultural products. While such residues may contain valuable materials, their current economic values are less than the apparent cost of collection, transportation and processing for beneficial use. Therefore, they are often discharged as wastes. During the manufacture process of the olive oil in mills, about 20% of oil, 30% of waste solids and 50% of wastewater are obtained (Baccar *et al.*, 2009). Olive-waste cakes (olive pomace), corresponding to the remaining residue of oil extraction process, represent a yearly average of 2x10<sup>5</sup> tons depending on the crop (Sellami *et al.*, 2008). The ashes of the olive-wastes can be considered as good adsorbents to get rid of the oxidation products of frying oil.

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The principal goal of the present study was to use ashes of olive wastes and to compare the adsorption efficiencies of this material with the already used synthetic adsorbents to regenerate the fried oil quality.

## Materials and Methods

### Materials:

Olive-wastes obtained from an oil factory located in Horticultural Research Institute, Agricultural Research Center, Giza, Egypt. Magnesol XL (Hydrous, White, amorphous and odorless synthetic magnesium silicate was obtained from Magnesol Product Division, Reagent Chemical and Research, Inc. Houston, Texas, USA. Sunflower oil was obtained from Savola Sime Company (Ramdan City, Sharkia Governorate, Egypt). Peroxide and acid values of the fresh sunflower oil were determined according to A.O.A.C. (2005). The corresponding values were 0.30 meq. peroxides/ kg oil and 0.12 mg KOH /g oil. These low values indicate the good quality of fresh sunflower oil. All chemicals used in the current study were obtained from Sigma Chemical Company (England, Landon, Ltd., Pools).

### Methods:

#### Preparation of olive waste ash:

Olive waste ashes were prepared according to the Method of A.O.A.C. (2005). The olive wastes were ground in grinder (Perten, USA) to a particle size of about 180nm, placed in a crucible, carbonized in air ashes in a muffle furnace (Furnace 5000; Thermolyne, IOWA, USA) at 600°C for 12h and then cooled to room temperature in a desiccator and stored in glass screw-capped bottles.

#### Metal content of ash:

The metal content of olive waste ash was determined using a plasma spectrometer ICP OES-Perkin-Elmer Analyzer by TB/TL/04/04 technique for (Si, Ca, Al, Mg, Mn, and Cu) while Fe was estimated by IB/TL/08/04 technique (Gil *et al.*, 2011).

#### Frying protocol:

A known amount (2 kg) of refined sunflower oil was placed in a domestic stainless steel pan fryer (60cm diameter X 30cm height), and heated at 180°C ± 5°C. Then, potato chips (2mm thickness X 40mm width X 50mm length) previously soaked in a sufficient quality of NaCl solution (10% w/v) to cover the chips for 10 min., were fried for about 10 min. The frying process was conducted four times each day, and the frying period was 4h/day. The weight of chips to be fried for each batch was about 200g. This process was repeated for six consecutive days, and the total continuous frying period was 24h. Oil samples were left to cool down and then stored at -5°C for analysis.

#### Treatment with adsorbents:

The adsorbent materials (olive-waste ash and Magnesol XL) were individually added to the used sunflower oil at 2% level, then mechanically stirred using a super -mixer (Gmmy Industrial Crop, Japan) for 15min. at 150 rpm, and filtered through Whatman No.1 filter paper. This adsorption treatment was repeated three times at the end of the entire heating period (24h) using new samples of used sunflower oil and filter aid.

#### Analytical methods:

##### Percent of free fatty acid (FFA):

FFA was determined using the A. O. A. C. (2005) method. Thirty milliliters of ethanol were used to dissolve 5g of the sample and titrated against 0.1N potassium hydroxide solution using phenolphthalein as indicator. FFA was expressed as % oleic acid.

##### Peroxide value (PV):

The PV was determined using the A. O. A. C. (2005) method. A known weight of oil samples (5g) was dissolved in a mixture consisted of glacial acetic acid: chloroform (30ml, 3:2 v/v) then freshly prepared saturated potassium iodide solution (1ml) was added. Distilled water (30ml) was added then titrated slowly with sodium thiosulphate solution (0.1 N).

*Induction period (IP):*

IP was measured using a metrohm 679 Rancimat (Metrohm AG, Herisau, Switzerland) following the (Firestone *et al.*, 1991).

*Viscosity:*

Viscosity was determined as Saguy *et al.*, (1996) using Brookfield DVIII model digital rheometer at 30°C using LV2spindle at 10, 20 and 50 rpm.

*Color index (CI):*

CI was determined as Nielsen (1998), measured with a Lovibond Tintometer Apparatus (The Tintometer Ltd., Salisburg, England) using an oil sample of 5 ml and an optical path length of 1. The yellow glass slides were fixed at 35 and the intensity of red glass was assigned through matching with the oil samples.

*Total polar materials (TPM):*

TPM were determined in oil samples using the Walkling and Wessels (1981), measured by column chromatographic.

*Polymer content (PC):*

PC was determined according to the method of Wu and Nawar (1986). One gram of oil was added to methanol (125ml) containing 1% H<sub>2</sub>SO<sub>4</sub>. The mixture was boiled under a reflux condenser for 2h and cooled to room temperature. The methanol insoluble were filtered and washed with methanol until no sulphuric acid remained. The washed insoluble polymers were dissolved in petroleum ether (25ml) and transferred to a pre weighed flask. The solvent was then evaporated under a stream of nitrogen and the flask was again weighted.

*Data analysis:*

At least three replications for each oil sample were performed with each test. The averages and standard deviation were calculated by statistical analysis using SPSS program 10.0 (IBM Corporation, Armonk, Ny). The differences were considered level significant when  $P < 0.05$  at a confident level of 95%. Arrangement of data for statistical analysis was performed by using Microsoft Office Excel (2007).

**Results and Discussion***Metal content of Magnesol XL and olive waste ash:*

Table 1 shows the metal content of Magnesol XL (synthetic adsorbent material) and olive waste ash (Agricultural wastes as a natural adsorbent). All adsorbent material under study was characterized by high levels of Si and variable levels of other metals. The adsorbent materials can be distinguished by their metal pattern. The highest level of Si was recorded for Magnesol XL followed by olive waste ash. The basic metals of Magnesol Si + Mg + Mn, while olive waste ash Si +Mg + Ca, respectively. These results are agreement with Farag *et al.*, (2009); Baccar *et al.*, (2009).

**Table 1.** Metal content of Magnesol XL and olive-waste ash.

Minerals (ppm)	Magnesol XL	Olive-waste ash
Si	360.00±7.30	325.00±6.85
Mg	294.00±5.10	261.00±4.55
Ca	140.50±2.30	255.00±4.60
Fe	8.30±0.33	7.30±0.29
Al	8.00±0.31	12.50±0.80
Mn	495.00±9.30	250.10±4.48
Cu	9.30±0.45	15.30±1.00

The data values ± SE are the mean values of three measurements for the same sample.

*Quality of fresh sunflower oil:*

Figures 1-7 show some physico-chemical properties of fresh sunflower oil. In general, the values of the physical and chemical properties for fresh sunflower oil quality under study (viscosity, color, free fatty acids, peroxide value, induction period, polar content and polymer content) were similar to those published by some researchers (Basuny and Arafat, 2014). These findings indicate that the fresh sunflower oil used was of good quality.

*Quality loss of fried sunflower oil:*

The changes in quality parameters of sunflower oil during intermittent frying are shown in Figs 1-7. It is worth noting that sunflower oil was heated at  $180^{\circ}\text{C}\pm 5^{\circ}\text{C}$  for 4h each day and this process was repeated for six consecutive days. Oil samples were taken at the end of each day and kept at  $-5^{\circ}\text{C}$  until analysis. Surveys have indicated that colour to be the main criterion for discontinuing usage of frying oil (Kajimoto, 1994). Regulations in several countries stipulate colour as a criterion for discarding frying oil (Firestone *et al.*, 1991). Colour development is an indication of oxidation, polymerization and formation of carbonyl compounds. It increases dramatically during frying and is influenced by frying temperature, although the quantitative amount of chemicals causing the increase in color is small (Paul and Mittal, 1997). The colour of fried sunflower oil samples collected at different periods was measured on a fixed yellow glass slide and variable red glass slides. The color of sunflower oil changed from clear yellow to dark brown especially during the last days of the frying process (Fig. 1).

The formation of polymers during frying is mainly responsible for the changes in the viscosity of the oil (Paul and Mittal, 1997). The viscosity of the frying oil is an indicator of oil sticking in large cavities of the crust of the food product. Higher viscosity results in larger volume of the oil in the food product (Paul and Mittal, 1997). Fig. 2 shows the viscosity of fried sunflower oil. The intermittent frying of sunflower oil led to a gradual and significant increase during the frying process.

Free fatty acid value is an indication of the degree of hydrolysis of oil. Controlling its level within a reasonable range of 0.4% (Berger, 1997) prevents acceleration of breakdown of fats, and also assists heat transfer. Free fatty acid values of sunflower oil increased significantly during frying and were strongly correlated with the heating time (Fig. 3). Hydroperoxides are the primary products of lipid oxidation. Therefore; the determination of peroxides can be used as an oxidation index for the primary stages of oil oxidation. The data in Figure 4 shows that the peroxide value of sunflower oil gradually increased with the frying time and became unacceptable after the first day of frying.

Induction period or so-called oxidative stability is an expression to describe the extent of oil stability by examines the time needed for oil to resist oxidation at elevated temperatures (Mhatthous, 2006). Resulted in Fig. 5 demonstrate a gradual decrease in the induction period of oils during the first stage of frying process.

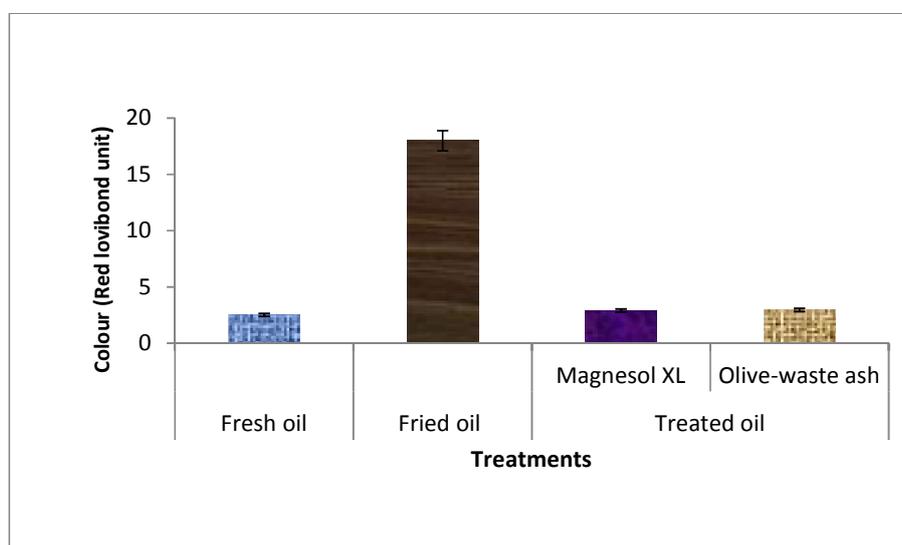
Quantification of polar compounds is considered as the most objective method to examine the deteriorative effect of frying oils (Mohamed Sulieman *et al.*, 2006). The polar compounds fractions that were polymerized and oxidized triacylglycerols and diacylglycerols and free fatty acids are being developed during the oxidation and polymerization stages (Dobarganes *et al.*, 2003). The polar content of sunflower oil under study was gradually increased during intermittent frying and there were significant differences in the contents of the polar compound (Fig. 6).

Polymer compounds which are a fraction of polar compounds are developed through tertiary oxidation and thermal modification in oil structure when exposed to high temperatures: the latter is more prominent based on the fact that steam release from the product provides some form of protection to the frying oil by minimizing contact with oxygen (Gertz and Kochhar, 2001). The formation of polymer compounds is responsible for the change in oil viscosity, tendency to foam during frying and imparts bitterness to the fried product (Samah and Fyka 2002). Furthermore, viscosity would also cause a considerable amount of oil adhering onto the product surface and thus increase the oil content (Maskan and Bagci, 2003). The development of polymer compounds across throughout the course of frying is shown in Fig. 7. At the end frying, the percentage of polymer compounds in sunflower oil was the greatest (8.70%).

*Effect of adsorbent material on sunflower oil quality:*

The efficiency of adsorbent depends on the polarity, surface active sites, surface area, porosity, particle size, pH and moisture content (Zhu, *et al.*, 1994). The color of fried sunflower oil was 35 yellow and 18.00 red. The treatment with Magnesol XL at the 2% level caused a significant decrease of red-coloured glasses. The same finding was achieved when fried sunflower oil was separately treated with olive-waste ash (Fig. 1). The viscosity of fried sunflower oil at  $180^{\circ}\text{C}$  for 24 hr was 71.00 centipoises. It was explained (Chatzilazarou *et al.*, 2006) that the increase in oil viscosity during frying is due to the C-C and C-O-C bridges formed between the

fatty acids due to high thermal energy yielding various sized polymers. Treatment with Magnesol XL remarkably lowered the viscosity of fried sunflower oil. The treatment with olive-waste ash almost had the same effect on the viscosity of fried sunflower oil (Fig. 2). The results demonstrate that the treatment of fried sunflower oil with Magnesol XL at 2% level led to significant reduction in the levels of free fatty acids. The data for fried sunflower oil samples treated with olive-waste ash was in accordance with the results of Magnesol XL treatments (Fig. 3). Similar results were achieved by Dulger and Yilmaz (2013) who used zeolites in removing free fatty acids from hazelnut oil. The peroxide value of fried sunflower was 41.30. The treatment with Magnesol XL resulted in a significant decrease of the peroxide value of fried sunflower oil from 41.30 to 2.55 meq/kg and this value agreed with the legalization and regulation of oil quality as reported by Firestone *et al.*, (1991). The efficiency of various adsorbent materials (Magnesol XL and olive-waste ash) in lowering the peroxide value of the fried sunflower was approximately the same (Fig. 4). In general, the adsorbent materials can be used effectively to lower the peroxide value of fried oils. Fried sunflower oil had an induction period of 2.00hr. Treatment with Magnesol XL significantly increased the induction period from 2.00hr to 6.30hr. Treatment with olive-waste ash induced the same effect on the level of induction period (Fig. 5). Fried sunflower oil had a polar content of 20.10%. Treatment with Magnesol XL significantly lowered the polar content to 2.9%, which agreed well with the oil quality regulations (IUPAC, 1987). Treatment with olive-waste ash significantly decreased and had nearly the same effect as that of Magnesol XL in reducing the levels of polar compounds (Fig. 6). The results demonstrate that the fried sunflower oil heated at 180°C for 24h had 8.60% polymers. The treatment of fried sunflower oil with different adsorbent materials at 2% level induced significant decrease in the polymer contents from 8.70% to 0.57 and 0.60 %. This indicates that the adsorbent materials under study had nearly the same adsorption efficiency in removing the polymers from the fried oil (Fig. 7). Overall, the data suggest that the adsorbent materials under study possess useful scavenging properties; they remove the oxidation products from the fried sunflower oil and the regenerated oil may be re-using in the food industry. In addition, the olive-waste ash had different levels of minerals. Generally, the present work provides information on improving the quality of fried sunflower oil using Magnesol XL and olive-waste ash at 2% level. The potential for using Magnesol XL as an adsorbent material to remove the oxidation products from frying oils is restricted by its irritant effect on skin and eyes. The natural adsorbent materials (olive-waste ash) do not exhibit any harmful effects on humans and possess nearly the same adsorbing effects in removing oil oxidation products. In addition, it is cheap and is useful for regenerating the quality of fried oils.



**Fig.1.** Changes in color of non-fried, fried and fried-treated sunflower with different adsorbent materials.

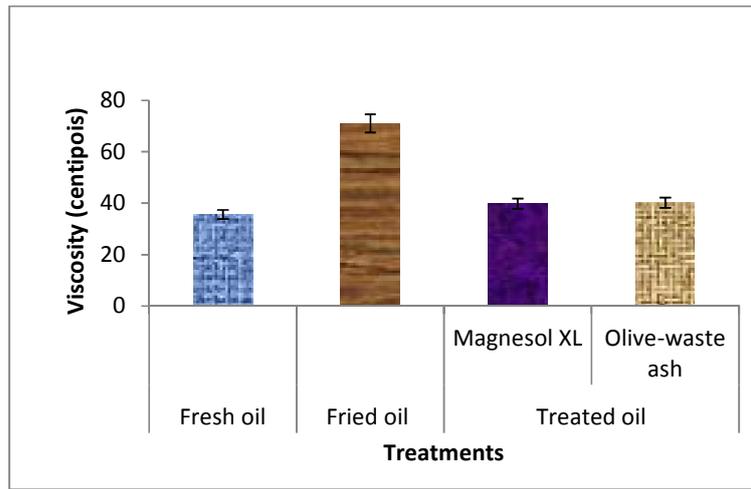


Fig. 2. Changes in viscosity of non-fried, fried and fried-treated sunflower with different adsorbent materials.

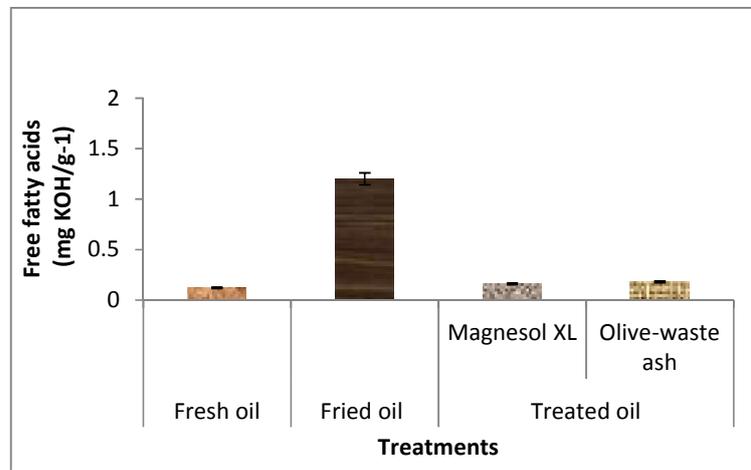


Fig. 3. Changes in free fatty acid (mg KOH/g-1) of non-fried, fried and fried-treated sunflower with different adsorbent materials.

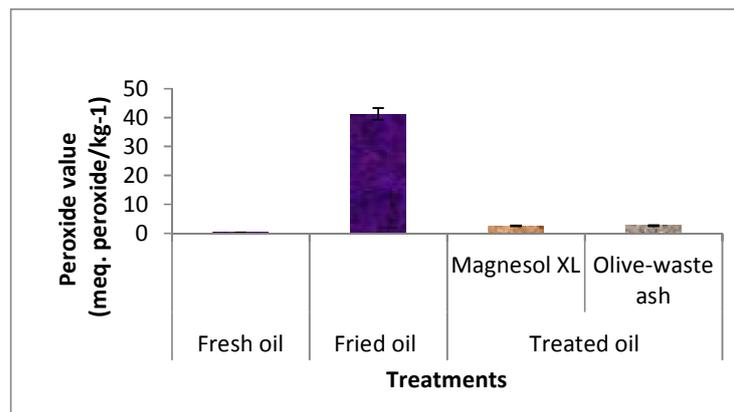


Fig. 4. Changes in peroxide value (meq. peroxide/kg-1) of non-fried, fried and fried-treated sunflower with different adsorbent materials.

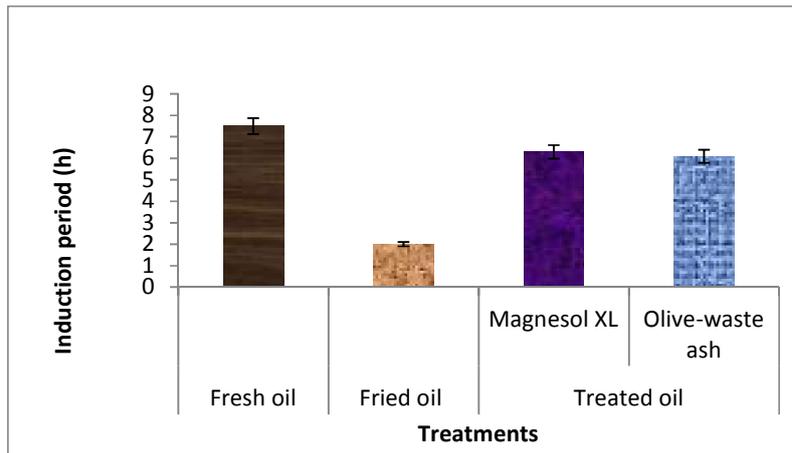


Fig. 5. Changes in induction period (h) of non-fried, fried and fried-treated sunflower with different adsorbent materials.

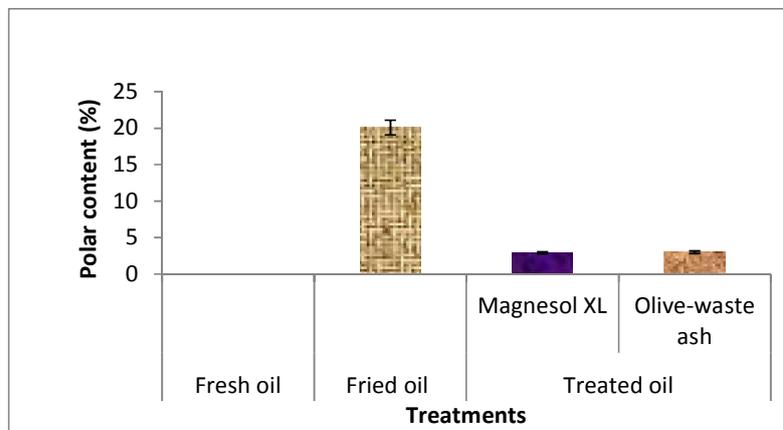


Fig. 6. Changes in polar content (%) of non-fried, fried and fried-treated sunflower with different adsorbent materials.

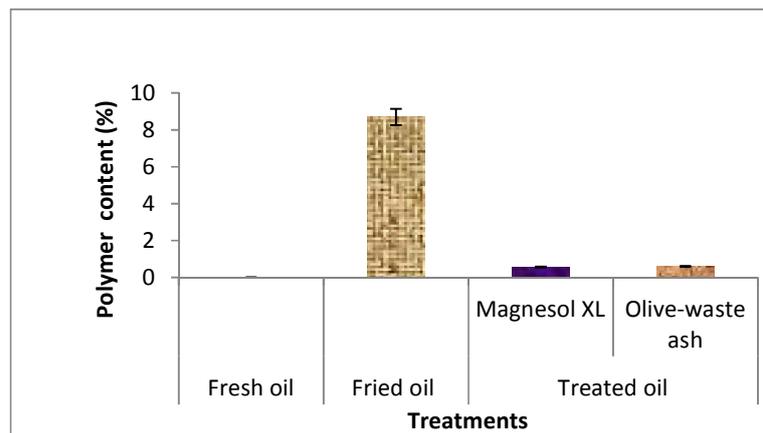


Fig. 7. Changes in polymer content (%) of non-fried, fried and fried-treated sunflower with different adsorbent materials.

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