American Journal of Chemical Engineering
2020; 8(1): 19-26
http://www.sciencepublishinggroup.com/j/ajche
doi: 10.11648/j.ajche.20200801.14
ISSN: 2330-8605 (Print); ISSN: 2330-8613 (Online)

A Simple Zeolite-based Treatment of Soya Bean Oil Mill Wastewater for Irrigation Purposes

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To cite this article:
Rose Erdoo Kukwa, Benjamin Ishwah, Ahola David Oklo, Donald Tyoker Kukwa, Fredrick Teghtegh Samoh, Aondoakaa Steve Nomor. A Simple Zeolite-based Treatment of Soya Bean Oil Mill Wastewater for Irrigation Purposes. *American Journal of Chemical Engineering*. Vol. 8, No. 1, 2020, pp. 19-26. doi: 10.11648/j.ajche.20200801.14

Received: January 25, 2020; **Accepted**: February 19, 2020; **Published**: March 10, 2020

Abstract: Soya bean oil mill wastewater (SOMW) is a liquid waste obtained from the soya bean oil industry with several environmental problems due to its high amount of toxic pollutants. This research work is aimed at assessing the feasibility and suitability of using a zeolite-based method for the treatment of soya bean oil mill wastewater for irrigation purposes. In this study, successive columns containing different types of solid-state materials were used to investigate the treatment efficiency of SOMW using physicochemical parameters; pH was determined using a pH meter, Turbidity determined using Turbidity meter. The concentration of Na^+, Ca^{2+}, Mg^{2+}, K^+ were determined using Flame photometer and the concentration of NO_3^-, SO_4^{2-}, PO_4^{3-} were determined using Oxygen Analyzer. Zeolite was characterized using Advanced Powdered X-ray diffractometer, energy dispersive spectrometer and Fourier Transformed Infrared and the fine sand characterized using an integrated X-ray Analyzer. The treatment columns were packed with fine sand, zeolite and zeolite/fine sand composite. The treatment decreased the concentrations of Na^+, Ca^{2+}, Mg^{2+}, K^+, NO_3^-, SO_4^{2-}, PO_4^{3-} and pH by mean percentages of 80.5, 29.6, 81.0, 2.1, 66.5, 41.4, 47.4 and 42.3%, respectively. The turbidity of the soya bean oil mill wastewater decreased by 72.5%. Most contaminants were removed in the soya bean oil wastewater in the zeolite/sand composite column. This decrease in the concentration of the pollutants could be attributed to the high sorption and ion exchange capacity of the solid-state materials used. This simple zeolite-based method is promising technology for the treatment of industrial wastewaters from oil processing industries for irrigational purposes.

Keywords: Zeolite, Soya Bean Oil Mill Wastewater, Physical Precipitation, Fine Sand, Column

1. Introduction

Nigeria is considered to be an agrarian State with tones of agricultural products such as; groundnut, soya bean, sorghum, maize and millet. They are produced yearly and later processed into edible oils thereby generating large amount of

Wastewater [1]. In most developing countries like Nigeria, wastewater obtained from these agricultural processing industries poses a major threat to humans, plants and aquatic lives when disposed into the environment without prior treatment [1]. Their treatment is thus essential before disposal [2]. The Vegetable oil effluent consists mainly of fats, oils, greases (FOG), nitrate, and sulphate and phosphates [3].

The processes that results to these industrial wastewaters from edible oil processing are known but not limited to degumming, deacidification, bleaching, hydrogenation, deodorization, winterinsing and the neutralization steps. Sodium salts of free fatty acid (soap stocks) are splitted through the use of sulphuric acid in the neutralization step, generating highly acidic and oily wastewaters. Its characteristics depend largely on the type of oil processed as well as the processing method, resulting in high amount of chemical oxygen demand (COD), oil and grease, sulphate and phosphate content, thus leading to high inorganic as well as organic loading of the relevant wastewater [4, 5]. The degumming operation, removes phosphatides. Some of the phosphatides in oils are readily hydrated hence water or steam is used to precipitate the phosphatides. The calcium and magnesium salts of phosphatides or lysophosphatidic acid are not hydrated by water but can be hydrated by alkali or acid [6].

Wastewaters from soya oil mill production are characterized by the following features and components: color ranging from intensive yellow to black, strong soya oil odor, high content of polyphenols (up to 80 mg/L), high content of solid matter (total solids up to 102.5 mg/L) and high content of oil (up to 30 mg/L). It is typically high in organic content, resulting in a biochemical oxygen demand (BOD) of 40-100 mg/L) and a chemical oxygen demand (COD) of 50-200 mg/L. The wastewater has a pH range of 4.5- 5.6, fat residues (50–100 mg/L), organic nitrogen (50–80 mg/L), and ash residues (40 to 50 mg/L) [7, 8]. Na^+, Ca^{2+}, Mg^{2+} and K^+ are the main ionic species present in the soya bean oil mill wastewater. In recent years, there has been increased attention directed towards finding the best methods to treat vegetable oil wastewater and toward recycling both the organic matter and nutritive elements for crop production [9, 10].

Various techniques have been developed for treatment of oil mill wastewaters. The treatment methods may be physical, chemical or biological in nature. Some of which include; solvent extraction, adsorption, chemical oxidation, foam filtration, filtration, ion exchange, sedimentation, membrane processes, stepwise coagulation, lime softening, coagulation, electrochemical processes, electrocoagulation, and chemical precipitation [11]. Most of these methods have drawbacks; including low efficiency for removal of trace concentration of pollutants in case of chemical/biological oxidation, electrolysis, ion exchange and solvent extraction [12]. Coagulation and precipitation processes produce large amount of sludge and require pH control [13]. Ozonation removes color from wastewater without decreasing the COD. Membrane processes suffer the problem of fouling of the membrane used [14]. Many of these processes lack in cost effectiveness with high energy intensive processing [15]. On the other hand, adsorption process has many advantages over the other processes. Some of which include; low operation cost, high flexibility, simple design and operation, easy automation, lack of sensitivity to toxic pollutants and the capability of operation at very low concentration, environmentally friendly, less investment in terms of initial cost [16]. Activated carbon had been the primary adsorbent of use over time because of its high capacity for removal of various pollutants. However, it brings with it the disadvantage of its high price and its high regeneration cost [17]. The use of zeolite is one of the most effective methods in wastewater treatment. Zeolites are environmentally and economically acceptable hydrated aluminosilicate materials with exceptional ion-exchange and sorption properties. They are naturally occurring crystalline aluminosilicate minerals that are derived from silica (SiO_2) by the isomorphous substitution of SiO_4- tetrahedra by AlO_4- tetrahedra. Recent investigations of natural and synthetic zeolites as adsorbents in water and wastewater treatment, their properties and possible modification of the zeolites have been a subject of many studies [18]. The use of zeolites in wastewater treatment has been reported by many researchers to be effective. In this present study, a simple zeolite low-cost method was used for Soya bean oil mill wastewater treatment. This was achieved by using successive columns of fine sand, zeolite and zeolite/sand composite.

2. Materials and Methods

2.1. Analytical Methods

Soluble Na^+, Ca^{2+}, Mg^{2+} and K^+ were determined using flame photometer (jenway PFP7). pH was determined using (pH 501 Eutech). Turbidity was determined using turbidity meter (NT-100). Zeolite Y was characterized using Advance powdered diffractometer (Bruker D8), FTIR and EDS, fine sand characterized using X-ray fluorescence spectrometer (ARL 9900 OASIS). Phosphate, nitrate and sulphate were determined using a direct reading spectrometer (JPB-607) portal.

2.2. Study Area

This research work was conducted in Makurdi town, the headquarters of Benue State, Nigeria. The town is located between latitude 7°38'N - 7°50'N, and longitude 8°24'E and 8°38'E and 104 meters elevation. It is situated in the Benue valley in the North Central region of Nigeria. It is traversed by the second largest river in the country, the River Benue.

2.3. Sample Collection

Twenty liters (20.0L) of fresh soya bean oil mill wastewater was collected (using sterile plastic container) at Seraph oil mills, Nigeria Limited, which is located at Km 7 Gboko Road Makurdi, Benue State. The soya bean oil mill wastewater was then transported immediately to Chemistry laboratory at Benue State University and refrigerated at 35°C prior to analysis. Fine sand was purchased at pillar poole Nigeria, limited. Zeolite Y was earlier synthesized in the Department of Chemistry Laboratory, Loughborough University, Loughborough, Leicestershire, United Kingdom.

2.4. Sample Digestion

The Environmental Protection Agency (EPA) vigorous digestion method described by Gregg (1989) was adopted. The sample digestion was done using nitric acid and heated until digestion completed. The sample was filtered and transferred into 100 mL volumetric flask and made to mark with distilled water. The wastewater digest was used for flame photometric analysis [19].

2.5. Physical Precipitation of Soya Bean Oil Mill Wastewater

Physical separation was carried out using glass cylinders with diameter 10 cm and length 20 cm. After 5 h of gravity separation, two kinds of suspended solids were observed: a floating part and a precipitated part. The floating part was decanted and the precipitate filtered using filter paper and a less turbid sample was obtained [20].

2.6. Soya Bean Oil Mill Wastewater Treatment Using Successive Steps

The soya bean oil mill wastewater (Soap stock) was passed through three designed treatment steps, following physical separation. Each treatment consists of successive column containing different solid-state materials. The columns were made of transparent glass material with an internal diameter of 2 cm and a length of 20 cm. The columns were sealed from the bottom using two pieces of gauze fabric firmly held by strings and tape. The columns were packed with solid-state materials to 15 cm height. The packing process was done in 5 cm increments to avoid segregation of particles. The columns were mounted vertically on a wooden holder in the laboratory. Each treatment system was replicated three times. The contact times between the soya bean oil mill wastewater and solid-state materials were 1 h for fine sand, 2 h for zeolite and 4 h for the fine zeolite/sand composite. The physicochemical parameters used to evaluate the treatment efficiency for each method were pH, phosphate, turbidity, sulphate, nitrate and metal ions concentration [20].

2.7. Characterization of Zeolite Y

Zeolite Y was synthesized and characterized using PXRD, EDS and FTIR. The PXRD used was a Bruker D8 Advance diffractometer operating with a copper X-ray tube, a monochromator with a Linx Eye detector. The PXRD data were collected using Cu Kα_1 (1.5406Å) radiation, over the 2θ range between 5 - 60° using a step size of 0.022° for 43 minutes. The EDS spectrum was produced on an EDAX Pheonix, EDX with a Carl Zeiss 1530 VP spectrometer. The samples were sprinkled onto 12mm aluminium stubs using "carbon sticky tabs". These were then gold coated using an Emitech SC 7640 gold/palladium sputter coater to reduce the static charges during the analysis.

The PerkinElmer paragon 1000 FTIR spectrophotometer was used to collect FTIR data for the sample. The sample was prepared by making discs of a small amount of the sample (~3 mg) in KBr and measurements were carried out over IR region of 1200-400cm^{-1} for the zeolite. A background spectrum was measured before the sample to compensate for atmospheric conditions around the FTIR instrument

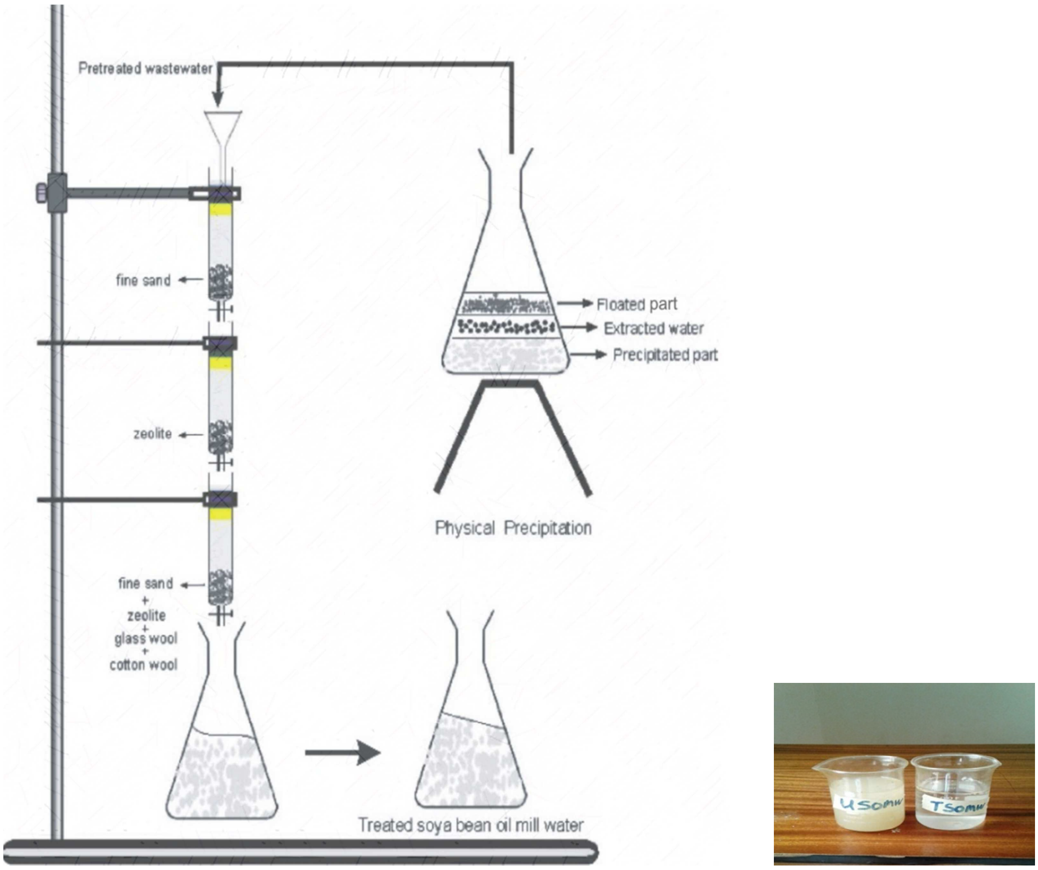

Figure 1. *Diagram illustrating the components of the soya bean oil mill wastewater (SOMW) treatment model and the appearance of the treated sample compared to untreated sample.*

3. Results and Discussion

3.1. Chemical Composition of Fine Sand

The chemical composition of the fine sand used in this present study was determined by X-ray fluorescence spectrometer (ARL 9900 OASIS Model) as shown in (Table 1). Silicon dioxide was found to be the predominant oxide, 92.49%. Other oxides present were Al_2O_3 7.44%, CaO 0.06% and MgO 0.09%. The silicon to aluminum ratio of the fine sand used was 12.40. This characterization data obtained is in line with earlier investigation reported [21]

Table 1. *Chemical composition of fine sand (XRF Spectrometer).*

Compounds/elements	% Weight
SiO_2	92.49
Al_2O_3	7.44
Fe_2O_3	ND
CaO	0.06
MgO	0.09
K_2O	ND

Key ND: Not detected

3.2. Synthesis of Zeolite Y

SEM-EDS

The elemental composition of zeolite Y was determined using energy dispersive spectroscopy (EDAX Pheonix model) as shown in (Table 2). The result revealed that zeolite Y consist of 48 7% Oxygen, 9.2% Sodium, 11.2% Aluminum and 30.7% Silicon. The Si/Al ratio (2.7%) calculated was found to be in good agreement with the data obtained from PXRD (2.4%) as calculated from the molecular formula of the zeolite Y ($Na_{54.91}Al_{56}Si_{136}O_{384}$).

Table 2. *Elemental composition of the synthesized Zeolite Y (EDS).*

Elements	% Weight
O	48.7±0.07
Na	9.2±0.04
Al	11.50±0.0
Si	30.7±0.1
Si/Al	2.7

PXRD

The PXRD data compared against the ICDD database for the theoretical phases showed good agreement that the targeted zeolite Y was formed as clearly seen from the matched patterns (figure 2). The PXRD patterns for the zeolite Y was a single crystalline phase with high degree of crystallinity.

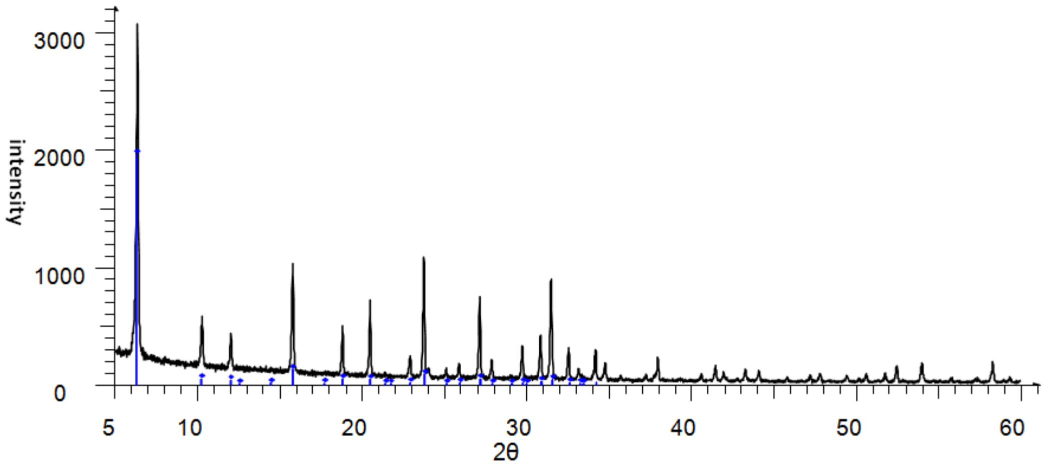

Figure 2. *PXRD pattern for zeolite Y (experimental pattern) matched with the reference pattern 01-070-4285 (vertical tick marks), anhydrous formula ($Na_{54.91}Al_{56}Si_{136}O_{384}$).*

FTIR

A broad band was observed in the region 1019 cm^{-1} which was attributed to the asymmetric vibrations of Si-O bridging and Si-O non-bridging bonds. The band in the range 717 cm^{-1} is due to the symmetric stretching of internal vibrations while the internal vibrations due to the bending of the T-O tetrahedra occurred between 455 cm^{-1}. Vibrations of the double six rings (D6R) connecting the sodalite cages occurred between 579 cm^{-1} while that around 626 cm^{-1} is assigned to the symmetric stretching of external T-O linkages in the zeolite. The band at 1150 cm^{-1} was attributed to the asymmetric stretching of external T-O linkages in the zeolite respectively [21].

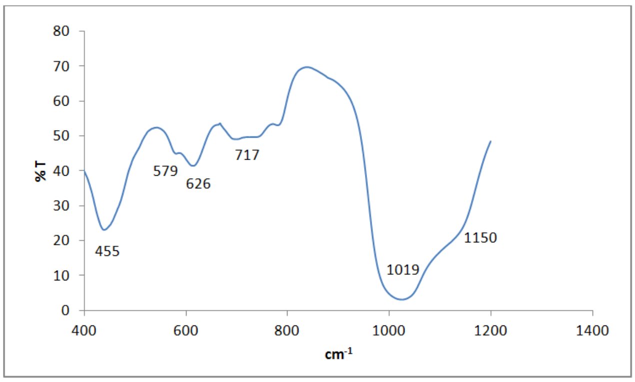

Figure 3. *FTIR spectrum for the synthesised zeolite Y.*

3.3. Characterization Results of Untreated Soya Bean Oil Mill Wastewater

The untreated soya bean oil mill wastewater was characterized by the following (table 3): pH 4.85, turbidity of 200 NTU, NO_3^- content of 35.80 mg/L, , PO_4^{3-} content of 4.7 5 mg/L *and* SO_4^{2-} content of 85.20 mg/L. The concentrations of Na^+, Ca^{2+}, Mg^{2+} and K^+ were 12.0703 mg/L, 10.5520 mg/L, 7.2833 mg/L and 10.5520 mg/L respectively. The results obtained were far above the maximum allowable concentration limits recommended for irrigation purposes by standard regulatory bodies like FOA (2010), EPA (2011) and WHO (2016). This implies that this soya bean oil water could not be used for agricultural purposes [22, 23]. Potassium was the predominant inorganic substance found in the untreated soya bean oil mill wastewater with a concentration of 15.5915 mg/L, it was considered been the main cause of SOMW salinity, similar results were obtained by some researchers [24, 25].

Table 3. *Average physicochemical characteristics of untreated and treated soya bean oil mill wastewater.*

Parameters	Untreated soya bean oil mill wastewater	Treated soya bean oil mill wastewater	FAO (2011)	WHO (2010)	EPA (2016)
pH	4.85±0.0	6.90±0.2	6.5-9.0	6.0-9.0	7.3-8.5
Turb. (NTU)	200±0.2	55.0	50.0	40.0	50.0
Ca^{2+} (mg/L)	10.5520±0.0	7.4026±0.1	5.0	6.0-7.0	4.5
K^+ (mg/L)	15.5915±0.0	15.2755±0.4	5.0-7.0	5.0	5.0
Mg^{2+} (mg/L)	7.2833±0.0	1.3815±0.2	2.0	2.0	2.0
Na^+ (mg/L)	12.0703±0.2	2.4241±0.1	6.0	2.0	3.0-4.0
NO_3^- (mg/L)	35.80±0.3	12.0±0.2	20.0	10.0	20.0
SO_4^{2-} (mg/L)	85.20±0.1	50.03±0.1	250.0	250.0	250.0
PO_4^{3-} (mg/L)	4.75±0.2	2.50±0.4	2.0	2.0-3.0	4.0

3.4. Soya Bean Oil Mill Wastewater Treatment Using Each Treatment Step

Physical precipitation

The treatment of SOMW by physical precipitation revealed a decreased acidity by 1.03%, Mg^{2+} concentration by 3.62%, turbidity by 12.50% SO_4^{2-} by 0.00%, PO_4^{3-} by 3.16% and NO_3^- by 15.08% respectively. The concentrations of the studied cations: Na^+, K^+, Ca^{2+} and Mg^{2+} decreased by 0.00, 0.12, 0.02 and 3.62% respectively. This could be attributed to the fact that the concentration of the cations and anions has been reduced through gravity settling, decantation and filtration [26].

Fine sand treatment

Fine sand treatment decreased the pH by 5.88%, turbidity by 24.57, SO_4^{2-} by 0.37, PO_4^{3-} by 12.39% and NO_3^- by 26.64% respectively. The concentrations of the studied cations: Na^+. K^+, Ca^{2+} and Mg^{2+} decreased by 41.67, 0.13, 22.18, 7.40% respectively. The decrease could be attributed to the fact that sand consist of 92.49% by weight SiO_2 and 7.44% Al_2O_3 given Si/Al of 12.4%. This clearly shows that in addition to ion exchange, the sand had the capacity to effectively adsorb these toxic substances, thereby decreasing their concentrations from the wastewater [27].

Zeolite Treatment

Zeolite treatment further decreased the acidity by 23.88% and turbidity by 39.39%. The concentration of the studied cations and anions such as Na^+ K^+, Ca^{2+}, Mg^{2+} SO_4^{2-} , PO_4^{3-} and NO_3^- decreased by 23.07, 0.77, 2.31, 68.00, 25.09, 16.88 and 35.43% respectively. The decrease in the toxic concentration of the wastewater could be attributed to the ion exchange capacity of the zeolite material [28].

Zeolite/sand composite

The zeolite/sand composite treatment (treatment 4) decreased the anions and cations concentration of Na^+ K^+, Ca^{2+}, and Mg^{2+}, SO_4^{2-} , PO_4^{3-} and NO_3^- by 55.35%, 1.29, 7.73, 33.65, 17.03, 25.37 and 16.66% respectively. The pH also decreased by 2.89 and turbidity to 1.23%. Most contaminants were removed from SOMW using treatment 3 and 4 (Zeolite and zeolite/sand composite treatment), this could be attributed to the high sorption and ion exchange affinity of zeolite material [28, 29]. Similar results were reported by some researchers on agricultural wastewater treatment using coagulation, flotation and chemical treatment [30].

Table 4. *Effects of the different treatment columns on soya bean oil mill wastewater physicochemical parameters).*

NO.	Treatment method	Physicochemical parameters after each treatment (Mean)								
		pH	Turb. (NTU)	Na^+ (mg/L)	Ca^{2+} (mg/L)	Mg^{2+} (mg/L)	K^+ (mg/L)	NO_3^- (mg/L)	SO_4^{2-} (mg/L)	PO_4^{3-} (mg/L)
1	Physical precipitation	4.8	175.0	12.07	10.55	7.02	15.61	30.40	85.20	4.60
2	Fine sand	5.1	132.0	7.04	8.21	6.50	15.59	22.30	80.50	4.03
3	Zeolite	6.7	80.0	5.42	8.02	2.08	15.47	14.40	60.30	3.35
4	Zeolite/fine sand	6.9	55.0	2.42	7.40	1.38	15.27	12.00	50.03	2.50

3.5. Treatment Effect on SOMW Using the Entire Successive Treatment

The soya bean oil mill wastewater treatment using successive columns containing solid state materials; fine sand, zeolite and zeolite/sand composite (table 4) decreased the toxic concentrations of Na^+, Ca^{2+}, Mg^{2+}, K^+, NO_3^-, SO_4^{2-}, $PO_4^{3-}$and pH by mean percentages of 80.5, 29.6, 81.0, 2.1, 66.5, 41.4, 47.4 and 42.3% respectively. The turbidity of the soya bean oil mill wastewater decreased by 72.5%. The predominant cation in the untreated soya bean oil mill wastewater is potassium which is believed to be the main cause of increased salinity [31, 32]. Using zeolite Y for desalination is considered a valid method for SOMW treatment since the toxic concentrations of Na^+, Ca^{2+}, Mg^{2+}, K^+ were greatly decreased. In this study, 41.4% of K^+ was removed by passing the SOMW through the zeolite/sand composite column, similar to the findings reported by some researchers [32]. The order of affinity of Zeolite Y to the alkali and alkaline earth metal cations is as follows: $K^+ > Ca^{2+} > Na^+ > Mg^{2+}$.

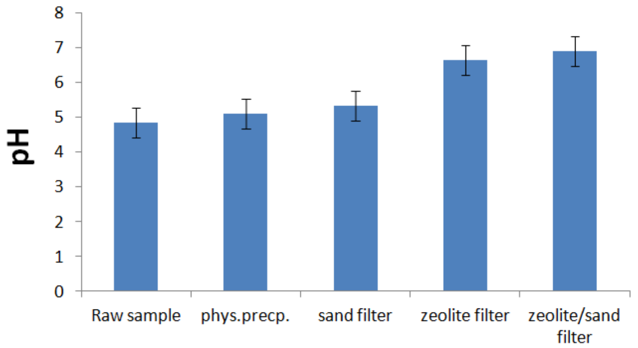

Figure 4. *Impact of different treatment steps on soya bean oil mill wastewater on pH*

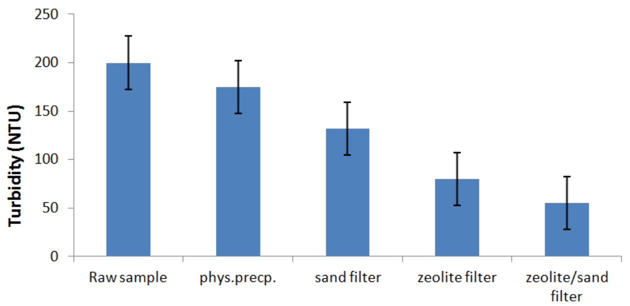

Figure 5. *Impact of different treatment steps on soya bean oil mill wastewater on Turbidity (NTU).*

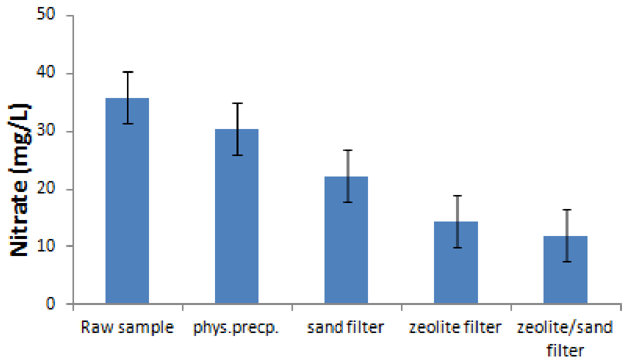

Figure 6. *Impact of different treatment steps on soya bean oil mill wastewater on Nitrate (mg/L)*

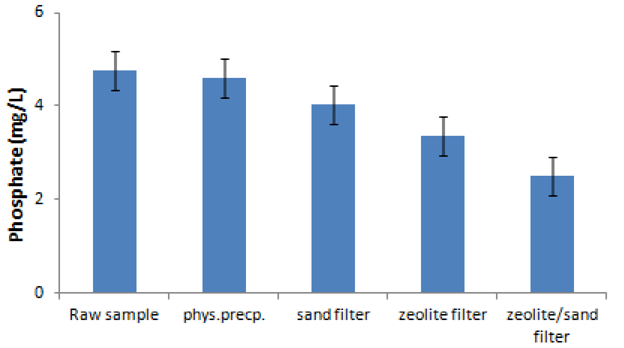

Figure 7. *Impact of different treatment steps on soya bean oil mill wastewater on Phosphate (mg/L).*

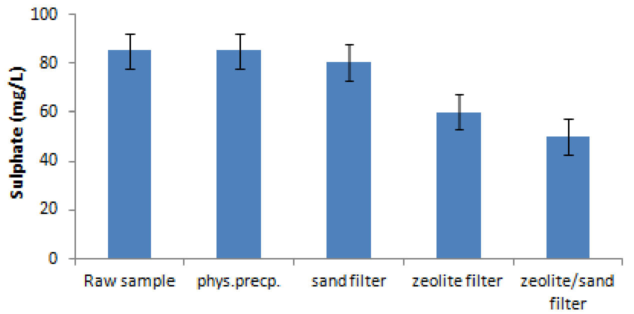

Figure 8. *Impact of different treatment steps on soya bean oil mill wastewater on Sulphate (mg/L).*

4. Conclusion

Several methods have been reported for Soya bean oil mill wastewater treatment; however very little research has been done on the treatment of soya bean oil mill wastewater using zeolite Y. In this present study, a simple zeolite low-cost method was used for Soya bean oil mill wastewater treatment. The treatment of soya bean oil mill wastewater using successive columns of fine sand, zeolite and zeolite/sand composite decreased the concentration of, pH, NO_3^-, SO_4^{2-}, PO_4^{3-}, Turbidity, alkali and alkaline earth metals such as Mg^{2+}, Ca^{2+}, K^+ and Na^+ which potentially increases soil salinity [33]. This method has proved to be effective in the treatment of soya bean oil mill wastewater.

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