Egyptian Journal of Aquatic Biology & Fisheries
Zoology Department, Faculty of Science,
Ain Shams University, Cairo, Egypt.
ISSN 1110 – 6131
Vol. 23(1): 233 -243 (2019)
www.ejabf.journals.ekb.eg

Treatment of aquaculture waste effluent to be reused in fish culture in Egypt

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ARTICLE INFO

Article History:
Received: Jan.4, 2019
Accepted: Feb.5, 2019
Online: Feb. 12, 2019

Keywords:
Aquaculture
Effluent
Reused
Environment
Adsorbent
Treatment

ABSTRACT

Aquaculture is currently the largest source of fish supply in Egypt. However, with this great productivity there has been an obsession with liquid waste and its impact on the environment. The aim of the present work is to make a preliminary trial to evaluate the ability of some low cost materials (sawdust, sawdust ash, granite powder, and crushed granite) to improve the quality of waste effluents resulting from recirculation aquaculture systems, and selecting the most efficient adsorbent to be applied at three different levels to verify which level is the most probable for waste effluent to be reused in fish culture. Total dissolved solids (TDS), electric conductivity (EC), salinity, dissolved oxygen (DO), turbidity, florin (F), chlorine (CL), phosphate (PO_4), sulphate (SO_4), ammonium (HN_4), nitirate (NO_3), nitrite (NO_2) were the evaluating parameter. Results from the adsorption experiment showed that the crushed granite was a promising adsorbent for aquaculture wastewater treatment. Accordingly, the effect of using different doses (5g/L, 10g/L and 20g/L) of crushed granite on water quality parameters was assessed and surface characteristics of cracked granite were determined using FTIR spectrophotometer for surface functional groups identifications, and X-ray diffraction (XRD) for mineralogical composition and nitrogen absorption for surface porous structure and surface area. Results revealed that crushed granite composed mainly of quartz and that 10g/L of crushed granite is the most probable level to be used with waste effluents for recirculation aquaculture systems.

INTRODUCTION

Water is a vital need for humans and all living organisms on earth. It is thus mandatory to preserve this important resource. Water quality is a key parameter in aquaculture industry. Fish is an inexpensive source of protein and an important cash crop in many regions of world (Bhatnagar and Devi, 2013). Optimum fish production from aquaculture majorly depends on integration between feeding and aquatic systems. Success in feeding system depends to a large extent on supplemental feeding (El-Sayed, 1999). At the same time, success in aquatic system depends on sufficient supply of water with proper physiochemical quality.

Phosphorus is one of the supplemental feed ingredients that are essential in fish diet, yet phosphorous in aquaculture effluents is considered a source of pollution by some of the regulatory agencies (Lall, 1991).

Uneaten ration and unavailable dietary phosphorous in feces are the two primary contributors in phosphorous effluent from aquaculture ponds. Most of the current fish production practices in Egypt are carried out without recirculation or treatment of effluents prior to its disposal (Ghanem and Haggag, 2015). Conventional excavated earthen aquaculture farms in the northern Nile Delta are reported to cause increase in nutrients (nitrogen and phosphorus) and organic wastes, through the feeding inputs, leading to general deterioration of water quality (Sipaúba-Tavares *et al*., 2013; Sipaúba-Tavares and Santeiro 2013). According to Ghanem and Haggag (2015) 14250 ton /year Ammonia, 10688 ton/year nitrate and 404 ton/ year phosphorous are discharged to the Mediterranean Sea from Kafr El-Sheikh alone. The cost of the environmental impact due to water pollution is relatively high with serious threatens to human health; increasing the severity of water scarcity problems, affecting the balance of aquatic ecosystems, economic development and social prosperity (Zyadah, 1996). Many attempts have been made to use different sorbents like activated carbon and alumina (Magriotis *et al*., 2014), yet the application of both adsorbents is limited by their high cost. Natural zeolites were tested and were proven to have a high affinity to metal ions yet it was found that natural zeolites needed to be modified before being used (Lemić *et al*., 2006; Motsi *et al*., 2009; Motsi, *et al*., 2011; Bao *et al*., 2013; and Itskos *et al*., 2015). Lakovleva (2018) stated that the final cost of zeolites is about 400 -500 USD per metric ton. Scientific researchers have searched for low cost-materials to be used for the treatment of polluted water like rice straw (Hegazy, 2008; and Ghanem and Haggag, 2015) cement klin dust (Hegazy *et al*., 2007). However, there are still some disadvantages concerning the known adsorbents. According to Gu *et al*., (2013) reported that bricks can remove phosphate and ammonium from aqueous solution with maximum adsorption of 3.8 mg/g for phosphate and 0.7 mg/g for ammonium. They have added that, it has rarely reported using one material to adsorb phosphate and ammonium simultaneously from aqueous solutions.

The adsorbents chosen for the present work are two locally available by-products in Egypt with expect adsorption capacity owing to their composition. Granite is a general term for a type of igneous rock composed of a variety of minerals including quartz, potassium and plagioclase feldspars, crystalline iron-bearing minerals (commonly amphibole and magnetite), and micaceous minerals with distinguished adsorptive capacities (Jan *et al.*, 2008). Egypt is ranked the fourth respectively with a share of 4.3% and 6.6% of total world market of marble and granite (Hamza, *et al.*, 2011). Sawdust is an abundant by-product of the wood industry that is either used as solid fuel for cooking or as packing material. It contains various organic compounds (lignin, cellulose and hemicellulose) with polyphenolic groups that create many active sites for adsorption (Rafatullah *et al.*, 2010)

The aim of the present work is to make a preliminary trial to evaluate the ability of some low cost materials (sawdust, sawdust ash, granite powder, and crushed granite) to improve the quality of waste effluents resulting from recirculation aquaculture systems, and selecting the most efficient adsorbent to be applied at three different levels to verify which level is the most probable for waste effluent to be reused in fish culture.

MATERIALS AND METHODS

Experimental system and fish

Mono sex Nile tilapia *(Oreochromis niloticus)* fingerlings were brought to Fish Experimental Unit in Regional Center for Food and Feed, Agriculture Research Center, Ministry of Agriculture, Giza, Egypt, from a fresh water commercial farm in Damietta Governorate. The experimental work was carried out in accordance with the national guidelines for care and use of laboratory animals and fish had received the utmost human care.

One hundred and thirty two mono sex Nile tilapia fingerlings *(Oreochromis niloticus)* of mean initial body weight 24.32 ± 0.31g from the stocked fish were randomly distributed into a 12 closed water recycle system (120 liter tanks), where each tank contained 11 fingerlings. The first 15 days of the experiment were considered as habituation period and thereafter the growth trials were carried out for 60 days. Diets (approximately 32 % crude protein and 17.09 MJ estimated gross energy) were randomly assigned to the experimental units. Fish were hand fed the experimental diets at 3 % of body weight for six days weekly, three times per day (Jauncey and Ross, 1982 and Coche, 1982).

The water flow rate out of each aquarium was 2L/min into a submerged biofilter after passing through a mesh net to remove solid impurities. Water was then collected in a common reservoir from which the filtered water was pumped up to the rearing units. Water used in the system was tap water stored for 24h to be sure that water is free of chlorine. Water was continuously aerated by a blower. Five percent of the total water volume was renewed in the early morning to compensate the loss in water volume resulted from evaporation. A thermo-controlled electric heater was used to adjust water temperature about 24±1ºC. All the experimental treatments were conducted under an artificial photo period equal to natural light/darkness period (12h light:12h darkness).

Waste water samples

At the end of the growth trial eighteen wastewater samples were collected in 0.5L bottles for each sample from the biofilter tanks using the technique proposed by Bradley *et al*., (2016). Three samples were transferred directly using thermally insulated container to water analysis lab and stored in refrigerators at approximately 4 ºC. The other fifteen samples were directly conducted with five different adsorbents (each three samples represent a treatment).

Adsorbents

The tested adsorbents included: Zeolite, sawdust, sawdust ash, granite powder and crushed granite. Zeolite extra pure synthetic was purchased from Sigma-Aldrich. Sawdust was obtained from local furniture factory. Sawdust ash was obtained by combusting the sawdust at 500°C for 4h in muffle furnace. Granite powder and crushed granite were obtained from Shaq Al Thobaan.

Wastewater treatment experiments

Batch experiments (15 samples) were conducted by adding 1g of each adsorbent to 100 ml (level 10g/l) wastewater in separate conical flasks and the flasks were capped. Flasks were then placed in a shaking water bath set at 100rpm at room temperature and left overnight. The water samples were then filtered and the adsorbents were removed. The samples were then subjected for physiochemical analysis.

The same batch experimental steps were then repeated using the most efficient adsorbent at three different levels (5g/l, 10g/l and 20g/l). Each level was done in triplicate.

Wastewater analysis

Standard Methods for the Examination of water and west water (APHA, 2005), were used to determine aquaculture recirculation wastewater quality characteristics (pH value), turbidity value (NTU), electrical conductivity (EC) value (µs/cm), total dissolved solids (TDS) (mg/L), salinity (mg/L) and dissolved oxygen (DO) (mg/L).

Inorganic Anions (Phosphate, sulphate, chlorine, nitrate and nitrite concentration (mg/L) were determined according to (USEPA 300.1, 1997 & USEPA 300, 1993) by using Thermo Scientific DionexICS-5000$^+$ Ion Chromatography supplied with Dionex Ion Pac AS23 Analytical column protected by Dionex Ion Pac AS 23 Guard Column for anions determination.

Ammonium (NH_4) and florin concentration (mg/L) were determined according to (ASTM D6919-03, 2003) and by using Thermo Scientific DionexICS-5000$^+$ Ion Chromatography supplied with DionexIonPacCS12A Analytical column protected by Dionex Ion PacCS 12A Guard Column for cations determination.

Adsorbent characterization

The different surface characteristics of cracked granite were determined using FTIR spectrophotometer (Thermo Scientific, USA), X-ray diffraction (XRD) for mineralogical composition and nitrogen absorption procedure by Brunauer–Emmett–Teller (BET) method for surface porous structure and surface area.

Statistical analysis:

Data are presented as means ± standard error (SE). The obtained data were subjected to one way analysis of variance (ANOVA) using the linear model (GLM) of SAS (SAS institute, 1991). Then means are compared adopting Duncan's new multiple range test ($P<0.05$).

RESULTS AND DISCUSSION

The physiochemical results of recirculation aquaculture effluent and the effect of the five adsorbent tested materials are shown in Table (1). The data revealed that the untreated recirculation aquaculture waste effluent has low water quality compared with desirable and acceptable range stated by Bradley *et al*., (2016) in term of dissolved oxygen (DO) and for desirable limit in term of nitrite. As the dissolved oxygen (DO) was significantly low (2.66 mg/l) compared to permissible value (DO)> 5mg/l. while, nitrite was higher (0.19 ppm) than desirable range (0 – 0.1 ppm).

All tested adsorbents had significantly decreased Turbidity, ammonia and nitrate load and improved the DO, compared to untreated waste effluent (Table 1). Sulphate and nitrite load were effectively decreased using different adsorbents except sawdust ash when compared to untreated waste effluents. Florin level was not affected by different treatments. Yet, in overall the statistical analysis of the different parameters shows that crushed granite significantly improved the studied waste effluent quality compared to the other adsorbents. Crushed granite and sawdust ash were the only two adsorbents to achieve the permissible value of DO (5.87 and 5.09 mg/l respectively). Dissolved oxygen (DO) is a very basic requirement for aquaculture species. It is usually the first limiting factor to occur in pond culture. The very low levels of DO are lethal after long term exposure; and at lower dissolved oxygen, only small fish can survive short-term exposures. For the cultivation of Nile tilapia the recommended DO should be >5 mg/ L.

Table 1: Adsorbents effect on circulation aquaculture waste effluent physiochemical parameters.[1]

Parameter	Untreated waste effluent from bio filter tank	Zeolite	sawdust	Sawdust ash	Granite powder	Granite crushed	Desirable range	Acceptable range
TDS (mg/l)	692±9[d]	454±20[a]	522±25[b]	1301±50[e]	581±25[c]	567±28[c]	--------	--------
EC (µS/cm)	1422±40[d]	930±25[a]	1066±45[b]	2661±100[e]	1190±50[c]	1151±57[bc]	60-2000	30-5000
Salinity %	0.67±0.03[c]	0.50±0.03[a]	0.50±0.01[a]	1.40±0.02[d]	0.60±0.03[b]	0.57±0.02[b]	0 – 10%	
DO (mg/l)	2.66±0.035[F]	4.27±0.05[c]	3.60±0.1[e]	5.09±0.09[b]	4.00±0.1[d]	5.87±0.12[a]	> 5	
Turbidity (NTU)	112.70±3[e]	18.00±2[b]	23.67±1.5[c]	11.00±1[a]	40.00±2[d]	8.67±0.5[a]		
F (ppm)	0.218±0.017[a]	0.410±0.009[d]	0.312±0.006[c]	4.367±0.015[e]	0.970±0.01[e]	0.24±0.02[b]	0.1 -1.5	< 3.0
Cl (ppm)	72.22±0.66[c]	105±1[d]	58±1[a]	246±1[e]	65±1[b]	59±1[a]	60 -100	---------
PO_4 (ppm)	68±1[d]	97±1[e]	17±1[c]	1.65±0.05[a]	12±1[b]	2.26±0.11[a]	0.01-2.0	< 3.0
SO_4 (ppm)	41.29±1[e]	9.18±1[a]	14±1[b]	101±2[F]	27.54±1[c]	30.78±2[d]	0 - 1000	
NH_4 (ppm)	27±1[e]	22±1[c]	20±1[b]	15±1[a]	25±1[d]	24±1[d]		
NO_3 (ppm)	12±2[c]	5±2[b]	1.33±0.05[a]	2±0.05[a]	1.33±0.05[a]	1.33±0.05[a]	---------	< 90
NO_2 (ppm)	0.19±0.05[c]	0.15±0.05[b]	>0.0001[a]	0.24±0.01[d]	>0.0001[a]	>0.0001[a]	0.0 – 0.1	< 4.0

[1]Values are the mean of triplicate n=3, means with different superscripts letters in the same row are significantly different (P<0.05).

Crushed granite had high efficiency in decreasing the average value of turbidity (92.3%) followed by the other adsorbents. Turbidity in waters decreases the available light needed for photosynthesis by phytoplankton and lead to the growth of undesirable organisms. Turbidity can shield food organisms, clog filters, causes damage and fish stress. Because many suspended solids will settle out in ponds or canals, another major concern besides turbidity itself is the amount of suspended particles that can potentially settle out Sediments from highly turbid source water may fill ponds and canals within a few months. Sedimentation can also smother eggs of some species in ponds used for natural reproduction (Zweig *et al.*, 1999).

Sawdust ash followed by crushed granite was significantly able to decrease the average value of phosphate in circulation aquaculture waste effluent to 1.65 ppm and 2.65 ppm, respectively. According to Stone and Thomforde, (2004) the acceptable phosphate level in fish culture is <3 mg/l (Bhatnagar and Devi, 2013). Different authors stated that the accumulation of phosphorous in natural or artificial water bodies is considered to be major cause of eutriopcation which cause excessive growth of algae, as well as sharp decrease of dissolved oxygen and finally fish toxicity (Montalvo, *et al*., (2011) and Gu, *et al*., (2013). In this study both sawdust ash and crushed granite was efficiently able to remove phosphate from circulation aquaculture waste effluent with a removing capacity reached 97.6 and 96.7 percentage, respectively.

Crushed granite, granite powder and sawdust decreased both nitrate and nitrite load to 1.33 ppm and >0.0001 (the detection limit of the instrument) with average removal efficiency 88.9% and 100% respectively. Nitrite is formed due to bacterial conversion (*Nitrosommonas)* of ammonia. Nitrite under normal condition is converted to nontoxic nitrate.

In case nitrite concentration is high in water, brown blood disease occurs lead to fish suffocation despite adequate oxygen concentration in water (Ghasemi *et al*., 2016).

Sawdust ash lowered the average value of ammonia to 15 ppm with average removal efficiency 44.4 % followed by sawdust 25.9% and zeolite 18.5%. Crushed granite and granite dust had minor effect on ammonia removal efficiency (11 and 7%). This result differs from what have been established by Mumpton, (1985) how stated that zeolite was able to remove upto 97% of ammonia produced in aquaculture. This contradiction in zeolite effectiveness have been explained by Ghasemi *et al*., (2016) how stated that zeloite ion exchange efficiency is affected by many factors such as zeolite type, pore size and particle size, chemical composition of the contaminated water as well as other factors such as water salinity. According to Lemić, *et al*, (2006); Motsi, *et al*., (2009), Motsi, *et al*., (2011), Bao, *et al* (2013) and, Itskos *et al*., (2015); unmodified zeolites have poorer adsorption capacity than modified one. Lakovleva (2018) have stated that the cost of zeolite modification can vary from 400 to 500 USD per metric ton.

Based on the results of adsorption experiment and considering the availability and cost of the adsorbents, the crushed granite was chosen as a representative adsorbent for aquaculture wastewater treatment.

Accordingly, the effect of using different doses of crushed granite on water quality parameters was assessed and the results are given in Table (2).

Table 2: Effect of different doses of crushed granite on water quality parameters[1]

Parameter	Granite 5g/l	Granite 10g/l	Granite 20g/l	Untreated	PROBABILITY
PH	8.062±0.15^{a}	7.005±0.25^{c}	7.697±0.25^{b}	7.059±0.35^{c}	P<0.01
TDS	572.7±27^{a}	567±28^{a}	557.3±12^{a}	692±9^{b}	P<0.001
EC	1205±56^{a}	1151±83^{a}	1195±56^{a}	1422±40^{b}	P<0.01
Salinity	0.6±0.03^{a}	0.6±0.02^{a}	0.6±0.025^{a}	0.8±0.04^{b}	P<0.001
DO	4.22±0.24^{c}	5.867±0.125^{a}	5.280±0.25^{b}	2.663±0.35^{d}	P<0.001
Turbidity	10.5±0.55^{a}	9.00±0.5^{a}	10.00±0.5^{a}	77.00±3^{b}	P<0.001
F (ppm)	0.255±0.013^{c}	0.240±0.02^{b}	0.30±0.015^{d}	0.218±0.017^{a}	P<0.01
Cl	40.00±2^{a}	52.40±2.6^{b}	55.70±2.8^{c}	72.22±0.66^{d}	P<0.001
PO_4	17.75±0.89^{c}	2.257±0.11^{a}	7.360±0.4^{b}	68±1^{d}	P<0.001
SO_4	37.45±1.86^{b}	17.35±0.87^{a}	18.85±0.94^{a}	41.29±1^{c}	P<0.001
NH_4	12.56±0.63^{a}	12.55±0.58^{a}	12.77±0.63^{b}	14.12±0.71^{c}	P<0.001
NO_3	1.370±0.07^{a}	1.26±0.03^{a}	0.51±0.02^{a}	12±1^{b}	P<0.001
NO_2	0.19±0.01^{c}	<0.0001^{a}	0.05833±0.0015^{b}	0.1933±0.05^{d}	P<0.001

[1]Values are the mean of triplicate n=3, means with different superscripts letters in the same row are significantly different (P<0.05).

Table 2 shows that, different dose from crushed granite have nearly the same significant adsorbent efficiency regarding total dissolved solids (17.24-19.5 %), electric conductivity (15.3-19.05%), salinity (25%), turbidity (86.4-88.3%) and nitrate (88.6-95.75%). All tested adsorbents had significantly improved the DO with both 10 and 20 gram crushed granite per liter achieved the permissible value of DO (> 5 mg /L). Fig.1. illustrates that 10 g crushed granite per liter has significantly decreased the average value of phosphate in circulation aquaculture waste effluent by 96.7 % compared to 20 and 5 g / liter (89.1 and 73.9% respectively). Also 10 and 20 g crushed granite were significantly able to adsorb 58 and 54.3 % respectively of sulfate in circulation aquaculture waste effluent Fig. 2.

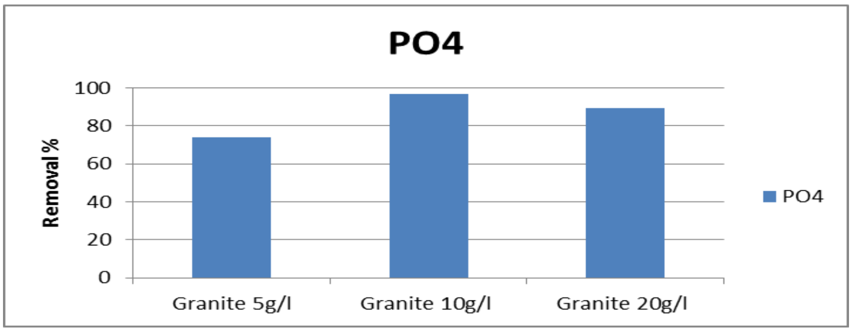

Fig.1: Effect of different doses of crushed granite on phosphate removal percentage.

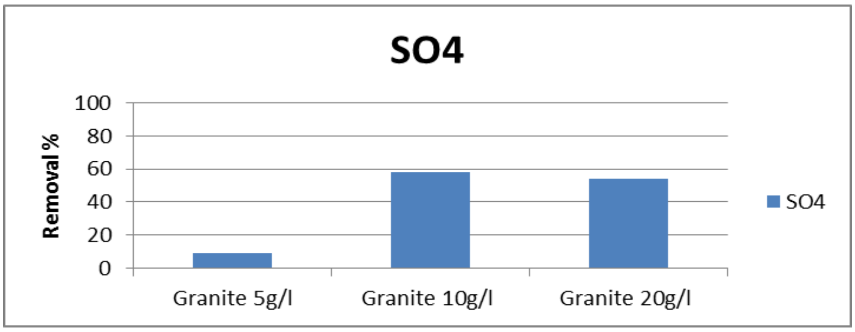

Fig 2: Effect of different doses of crushed granite on sulphate removal percentage.

Fig.3. shows the effect of different dose from crushed granite on nitrite, nitrate and ammonia. Ten gram crushed granite has significantly removed nitrite completely from circulation system waste effluent. Twenty gram crushed granite was able to remove 69.8% of nitrite, while five gram crushed granite was able to remove only 1.7% of nitrite. While different doses of crushed granite nearly have the same removing capacity of nitrate. On the contrary different dose from crushed granite have minor significant ammonia removal efficiency, with ten gram crushed granite per liter being the most efficient dose (11.1%). From the above results it is clear that the highest removal percentages were obtained at an adsorbent dose of 10g/l.

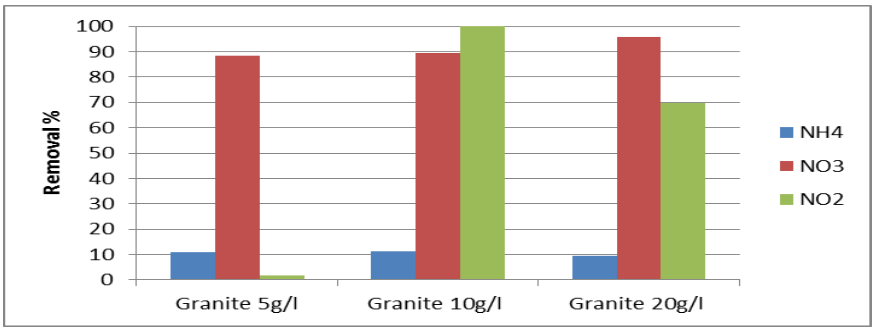

Fig 3: Effect of different doses of crushed granite on ammonia, nitrite, and nitrate removal percentage.

Crushed granite characterization

The crushed granite was characterized by different tools to get more info about its structure. The BET plot of crushed granite is given in Fig.4. The surface area, total pore volume and average pore size of the crushed granite were found to be

22.156 m^2 g^{-1}, 0.014 g cm^{-3} and 1.283 nm, respectively. The average Particle radius was also determined to be approximately 61.545nm.

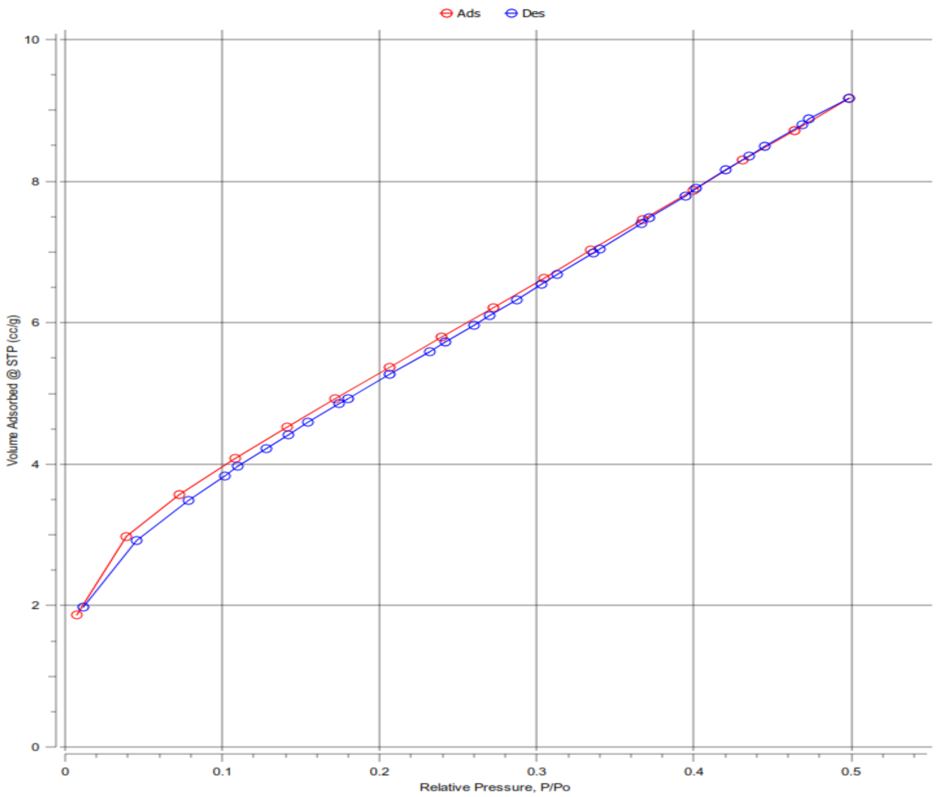

Fig 4. BET plot of crushed granite.

It is known that, the adsorption ability of an adsorbent is usually related to its own surface area, which depends on pore volume and size (Wu *et al.*, 2014). Thus the high surface area of crushed granite along with its pore volume and small size indicate its enhanced adsorption ability.

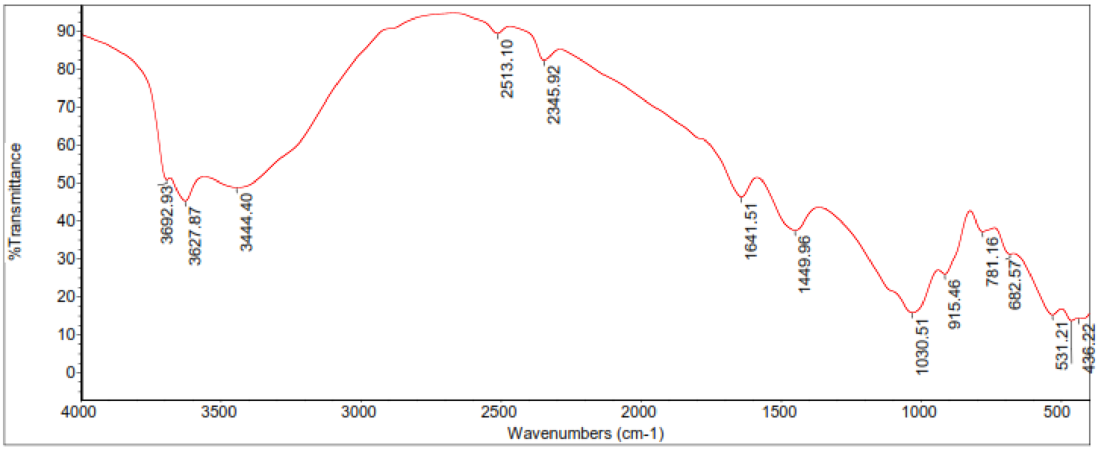

Fig. 5: FTIR spectrum of crushed granite.

The FTIR spectrum of crushed granite (Fig.5) shows that the region 781.16 - 436.22 cm-1 indicating the presence of quartz. Also, a peak at 1030.51 cm^{-1} can be assigned to characteristic bands of silicates which are mostly related to stretching vibrations of Si-O or Al-O (Aroke, Abdulkarim and Ogubunka, 2013). The peaks at 3680, 3580, and 3400cm^{-1} are due to OH bond stretching for Al-OH and SiOH. The band at 1030.51 cm^{-1} corresponds to the oxygen-bridging Si-O-Si asymmetric stretching and non oxygen-bridging Si-O stretching in granite (Aroke, Abdulkarim and Ogubunka, 2013). The presence of these functional groups of crushed granite could act as effective adsorption sites for contaminants, and thus increase its adsorption capacity. The presence of Silica groups on the crushed granite is very important for the adsorption especially that of phosphate ions. The phosphate groups can form strong hydrogen-bonded complexes with the silanols of the silica surface (Murashov and Leszczynski, 1999).

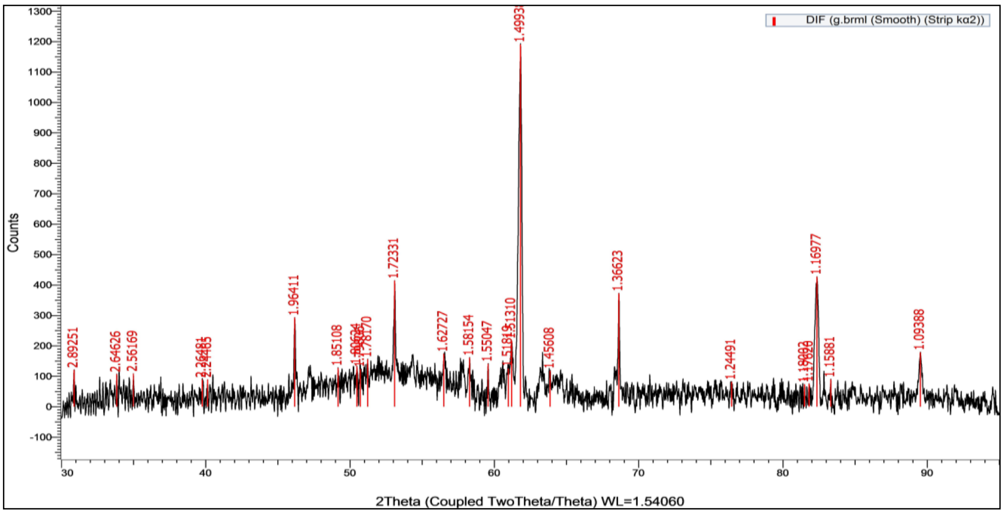

Fig. 6: XRD diagram shows the main content of crushed granite.

The XRD of crushed granite (Fig. 6) shows that the granite mainly contains quartz (SiO2) which is in accordance with previous studies showing that granite is mainly composed of quartz, calcite (CaCO3) , Al2O3 (alumina) and other minerals (Hamza *et al.*, 2011; El- Hinnawi and Abayazeed 2011 and Gao *et al.*, 2017).

CONCLUSION

According to the Egyptian strategy there is a continuous effort to develop wastewater management plans for aquaculture to protect the environment and the natural resources. In this study crushed granite gave promising results on its adsorbent ability on circulation aquaculture waste effluents with 10g/L being the most efficient dose. This will not only constitute an added value to aquaculture industry but will also lead to manage the undesirable residues from granite industry. More field studies are needed to see the effect of different environmental factors on crushed granite adsorbent capability.

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