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Optimization of pH and Contact Time of Media in Removing Calcium and Magnesium from Groundwater

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Abstract: Biosorption is one of the method which can be used to remove metals and pollutants from groundwater. This study has been conducted to optimize pH and contact time of media in removing the Ca2+ and Mg2+ in groundwater. The media that was used in this study are activated carbon made up from rice husks (RHAC), sands and zeolite. Media characterization using XRF, FTIR and SEM were used to identify the changes in media morphology. The method used in this study is the filtration process which will be done through batch study. In this batch study, the optimum condition of pH and contact time for removal of metals from groundwater are found to be at pH 7 and 60 minutes. All the media that were used in this study were found capable to remove the metals and pollutant in groundwater.

Keywords: Activated carbon, adsorption, sand, water filtration system, water hardness, zeolite.

1. Introduction

In Malaysia main source of water supply are from surface water, despite that there are several state in Malaysia use groundwater as an alternative source[1]. Unfortunately groundwater are associated with water hardness, since water moves through soil and rock and will dissolves small amounts of naturally occurring minerals and carries them into the groundwater supply.

Water is a great solvent for calcium (Ca^{2+}) and magnesium (Mg^{2+}), high concentrations of Ca^{2+} and Mg^{2+} ions were consider as hard water [2]. Potential health problems associated with hard water were kidney stones, dermatitis, re-productive health issues and pancreatic cancer [3]. Dissolved Ca^{2+} and Mg^{2+} are primarily responsible for most scaling in pipes and water heaters and cause numerous problems in laundry, kitchen, and bath. Hardness is usually expressed in grains per gallon (or ppm) as calcium carbonate equivalent. General guidelines for classification of waters are: 0 to 60 mg/L (milligrams per liter) as calcium carbonate is classified as soft, 61 to 120 mg/L as moderately hard, 121 to 180 mg/L as hard, and more than 180 mg/L as very hard [4].

Cation exchange (inorganic, carbonaceous, or organic base exchangers), membrane separation (nano filtration), precipitation using lime-soda and adsorption are common techniques for softening water [5]. Although ion exchange is

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very effective in use, it is an expensive treatment technique. However, from these mentioned techniques, adsorption is more advantageous in terms of economy, design and working flexibility, effectiveness, efficiency and high quality purified product [3]. Furthermore it is important that the adsorbents used in the adsorption technique can be used repeatedly after some of the applied processes. The results from previous studies revealed that activated carbon, biosorbents, carbon nanotubes, clays, metal oxides and zeolite have proved magnificent removal capabilities for most of heavy metal in water and waste water [6].

This study exploring three types of media namely sand, zeolite and activated carbon from rice husk. Mean of the material was analyzed using different machine to analyze different properties of the material. According to Jiang [7] sand is a stable silicate mineral that is widely used as a metal substrate and total suspended solid screening in water treatment. The study by Ahammed [8] for groundwater treatment showed that the effective size of sand used for continually operated slow sand filters should be in range of 0.15 – 0.35 mm, and that the uniformity coefficient should be in range 1.5-3 and coefficient of less than 2 is desirable. Sand must be washed before being used to ensure its free from any clay, soil or organic matter. In sand filtration there is complete physical, chemical and biological treatment in one unit [9].

Zeolite is one of the most widely used in drinking water treatment. Previous study have established that zeolite can removed anions and organics from groundwater systems [10]. Furthermore it was used as heavy metal removal due to the low-cost and bulk availability of raw material. The presence of well-defined molecular and porous structure, high thermal stability, ion selectivity, ion exchange capacity and surface area were among the factor which contribute zeolite as a suitable material which can be used effectively in hard water softening [11].

Activated carbon has been typically used as adsorptive material to remove organic compounds from water due to its unique textural properties of high specific surface area depending on the raw materials used for the production of activated carbon [12]. Recently, numerous approaches have been studied for development of cheaper and effective adsorbent. As a result of its high cellulose and lignin content, rice husk can be used as the raw material to prepare activated carbons, which are known to be highly complex porous structures, with high values of specific surface area and porous sizes mainly in the range of micropores [13]. The efficiency of rice husk as an activated carbon (RHAC) had been proved by Ushawary [14].

Table 1 - Filter media and filtration capability

Media	Remarks
Sand	Sand filtration unit can remove up to 99% of the bacteria and other unnecessary contaminants from the groundwater supply [15].
Rice husk activated carbon (RHAC)	RHAC adsorbents have proved magnificent removal capabilities for most of the heavy metal ions furthermore it will save disposal cost and alleviating potential environmental problems [16].
Zeolite	It is an effective sorbent and ion –exchange media for metal ions which was evaluated as an ion exchange media for the removal of heavy metals from acids mine waste water [17].

Therefore, from previous researches as in Table 1, sand, zeolite and RHAC were chosen in this study as media for groundwater treatment. These media have the potential to replace expensive conventional, softening treatment techniques as well as reduce the usage of excess chemicals (chemical precipitation) and it was used as filtration media, by using this media it will decrease cost for water hardness treatment. Groundwater will undergo the adsorption process as treatment mechanism.

The study of physicochemical and morphological characterization of sand, zeolite and RHAC is important to identify their adsorption efficiency and capacity. The physicochemical characterization using Scanning electron Microscope (SEM). SEM images will indicate the porosity of the sand and zeolite surface and also the honeycomb microstructure of RHAC.

The morphological characterization was determined by Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Fluorescence (XRF). FTIR will identify a surface functional group of all the media whereby functional group is a portion of a molecule that is classified group of bound atoms. In organic chemistry it is very common to see molecules comprised mainly of a carbon backbone with functional groups attached to the chain. The functional groups give the molecules its properties different functional groups were able to react with groundwater [18]. The XRF application is of utmost importance in order to confirm the existence of potential adsorbing element in sand, zeolite and RHAC. This technique was found useful in elemental analysis by previous researches [19].

Process of adsorption is influenced by some factors. There are temperature, pH, contact time, and initial concentration of adsorbates [18]. In this study, the batch experiment was conducted at room temperature using groundwater to study effect of contact time and pH of adsorption. The purpose of this study is to investigate the possible use of these media to reduce Ca^{2+} and Mg^{2+} in groundwater.

2. Methodology

2.1 Water Sampling

The groundwater samples were collected at a depth of 132 m below ground level located at Lot 1981, Bukit Tembaga Utara, Kedah. The samples are taken directly from the well which is equipped with pump during 9 month period from April 2016 until January 2017, the chosen period will cover all the climate condition, rainy and dry season and being collected at different time, morning, noon and evening. Every sample must be in triplicate and the result was from the average data. The preliminary data of groundwater on site were observed after the sampling is done.

2.2 Media Preparation

The preparation of activated carbon from rice husk with impregnation method with nitric acid and carbonization process. Impregnation is the process where rice husk being impregnated in nitric acid with 10% concentration. Nitric acid with low concentration were used as this water will be treated for drinking purposed [19]. This process will enhance the development of pore size for activated carbon [20]. The rice husk carbonized at temperature 450°C for 20 minutes. Carbonization process is the process which produces a distribution of interval pores and affects in carbon surface which is generally to enhance its absorptive capacity [12].

The raw sand was washed with distilled water and oven dried for 24hours to clean any dirt. The optimal size of sieve screen (0.7 mm opening size) was chosen in preparing the filtration sand. It provides the recommended effective size (ES) for the filtration sand (0.15 to 0.20 mm) the uniformity coefficient (UC < 2.5) and the maximum amount of very fine sand grains (maximum 4%< 0.1mm passing through #150 mesh screen) [21]. The chiloptilolite zeolite which 0.3 – 1.0 mm size in range were purchased from Syarikat Saintifik Bersatu. The raw zeolite were washed with distilled water and oven dried for 24 hours to clean any dirt.

2.2 Media Characterization

The study of surface chemistry of solids is possible by using X-Ray Fluorescence (XRF) Spectroscopy analysis The sample preparation involved sequence works; grinding the media, pulverizing the media less than 150 micrometer, weighed 8.0 g of the sample and added with 2.0 g binder to make a homogenously pellet. The PE-MAN Manual Hydraulic Press was used to form the mixture into pallet.

Fourier Transform Infrared (FTIR) spectrometer (PE spectrum 100, USA) was employed to determine the presence of surface functional groups in each media at characteristics wave numbers between 500 and 4500.

The surface morphology of the media before and after treatment was analyzed using a scanning electron microscope Philips, Quanta FEG 200 model (SEM) equipped with an EDX spectrometer from oxford instruments.

2.3 Batch Experiments

Batch study is the most suitable experiments for optimizing pH and contact time for metal removal in groundwater sample [3]. This experiment was carried out at different initial pH values. The range of pH of the metal ion solution was 6.0, 6.5, 7.0 and 7.5. The pH of solution was adjusted using sodium hydroxide and nitric acid to achieve the desired pH values.

All experiments conducted using a batch method in 250 mL conical flask. The conical flask were filled with groundwater (200 mL) and added with 4 g of sand, 3.6 g for zeolite, and 1 g for RHAC. All the samples were agitated at 125 rpm at room temperature [2]. The time interval was for every 15 minute which were 15 min, 30 min, 45 min, 60 min and 75 min. The biosorbent dosage was based on actual column weight. The experiment was repeated by varying the contact time of media with constant pH. The working condition for batch studies as tabulated in Table 2.

Table 2 - Working condition used for batch studies

Metal	Contact time (min)	pH
Ca2+	15, 30, 45, 60, 75	5.5, 6.0, 6.5, 7.0, 7.5
Mg2+	15, 30, 45, 60, 75	5.5, 6.0, 6.5, 7.0, 7.5

The metal ions (Ca^{2+} and Mg^{2+}) removal efficiency (RE) was determined using Eq. (1) [22].

$$R_e = \frac{C_{in} - C_o}{C_{in}} \times 100 \qquad (1)$$

where C_{inlet} = initial concentration of Ca^{2+} and Mg^{2+}, and C_{out} = Ca^{2+} and Mg^{2+} concentration after treatment. In order to ensure the reproducibility of results, all the biosorption experiments were performed in triplicate and the results are

presented as means of the replicates along with standard deviation (represented as error bars). Microsoft Excel 2007 program was employed for data processing [23].

3. Results and Discussion

3.1 Groundwater analysis

Table 3 presents the result for groundwater from study area. From the table it can be seen that only certain element in the groundwater are above the permittable value by WHO. Turbidity value is higher due to suspended sediments, such as particles of clay, soil and silts frequently enter the water from disturbed sites and affect water quality. It is important to note that higher turbidity level will give a sign that there is pollutant present in the water and also it will affect water clarity [24].

The total dissolve solid (TDS) show the content of anion and cations, such as calcium, magnesium, sodium, potassium, hydrogen carbonate, chloride sulfate and nitrate anion in groundwater. From the table the amount of Ca^{2+}, Mg^{2+} and potassium is the highest value for the groundwater at the study area, Mg^{2+} is higher than permittable value by WHO, this is due to water is a great solvent for calcium and magnesium and it will dissolves small amounts of naturally occurring minerals in the soil around the water source [25]. The water content of higher concentration of Ca^{2+} and Mg^{2+} is classified as hard water [3]. From this result Ca^{2+} and Mg^{2+} ions were chosen as the parameters of metals to be removed.

Table 3 - Groundwater result for study area

Parameter	Value in mg/L (unless otherwise	Drinking water standard (WHO,
Turbidity	120	0.1
Total dissolved solid	4	5
Dissolved oxygen (DO)	9	N
pH	6.	6.5-
Chloride (Cl^-)	32.	2
Sulfate (SO_4)	20.	2
Pottasium (K^+)	288	2
Calcium (Ca^{2+})	206	N
Magnesium ($Mg^{2+)}$)	450	30 mg/L

The result for chemical composition for sand, zeolite, and RHAC are tabulated in Table 4. The main chemical composition of zeolite and RHAC were SiO_2 (65.7%) and (78.79%) while for sand the highest chemical composition is CaO (54.71%). The chemical composition in every media indicates the content of cations. These cations are exchangeable with certain cations in groundwater such as lead, cadmium, zinc, and manganese, makes them particularly suitable for removing undesirable heavy metal ions from groundwater. This is in agreement with similar composition of sand, zeolite and RHAC as filter media for drinking water and waste water treatment [28], [29].

Table 4 - XRF analysis for sand and zeolite and RHAC

Chemical	Sand	Zeolite	RHAC
Silica ioxide (SiO_2)	0.77	65	78.79
Aluminium oxide	0.11	13	N
Ferric Oxide (Fe_2O_3)	0.36	1	1.
Sodium Oxide (Na_2O)	N	1.	0.
Pottasium oxide (K_2O)	N	3.	0.
Calcium oxide (CaO)	54.71	3.	2.
Magnesium oxide MgO	0.21	0.	1.
Sulfur trioxide (SO_3)	N	N	9.
TOTA	56.17	88.58	93.16
L	43.83	11.42	6.

3.2 FTIR Analysis

Fig. 1 to Fig. 3 present the FTIR result for RHAC, zeolite and sand. Peaks appearing in the FTIR spectrum were assigned to various chemical groups and bonds in accordance with their respective wavenumbers (cm^{-1}) as reported in Table 5 to Table 7. The FTIR spectra obtained were in agreement with the result report by Othman [28] for activated carbon produced from cucumis melo rind. Fig. 2 presents FTIR analysis result for zeolite. Band at 1650 cm^{-1} is attributed to the flexion vibration of OH group. Finally, a typical stretching broad band of zeolite is present around 3361 cm^{-1} same as RHAC.
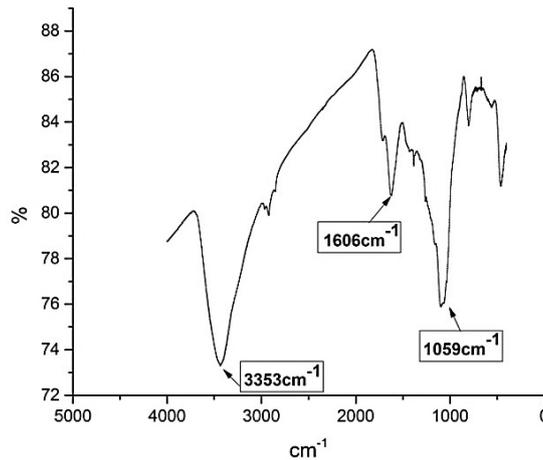
Fig. 1 - FTIR spectra of rice husk activated carbon with carbonization temperature 450°C for 20 minutes

Table 5 - The FTIR spectral characteristics of RHAC

Wavelength Range (cm⁻	Wavenumber (cm⁻	Identified group	Referenc
3500-3200	33	Bonded hydroxyl	
	53	group	
1640-1500		(-OH) and (-NH)	[2
	16		9]
1300 - 1000	1059	-C-O or –C-N bonds	
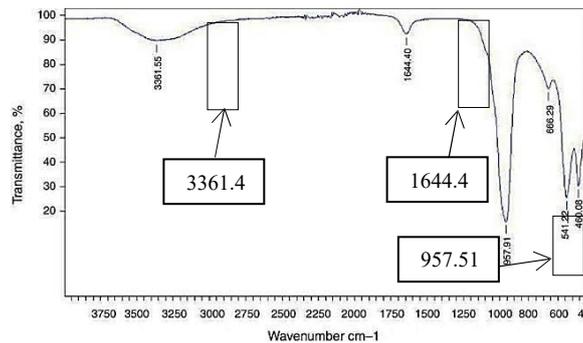
Fig. 2 - FTIR spectra of FTIR spectra of zeolite

Table 6 - The FTIR spectral characteristics of zeolite

Wavelength Range (cm⁻¹)	Wavenumber (cm⁻¹)	Identified group	Reference
3500-3200	3361.55	Bonded hydroxyl group (-OH) and (-NH) groups	[29]
1640-1500	1644	Carboxylate and carboxyl groups	
1060-895	957.91	Secondary cyclic alcohol	

Based on the FTIR analysis, it is indicated that presence of wide spectrum of functional groups in such alcohol, phenol and carboxylic group. This analysis concluded that all media produced different band spectra will give different adsorption intensities due to different surface functional group.

From Fig. 1 and Fig. 2, a similar spectrum of FTIR for zeolite and sand which indicate that surface adsorption and ion exchange as the driving forces of ion removal in hydroxyl groups. According to Liu [31] the carboxylic group has the highest affinity towards water molecules, this was attributed to the perfect match for hydrogen bond formation between the water molecule and the carboxylic group.

Based on Fig. 3 the identified group for sand were similar with research by Guchi [32] which indicate that sand is suitable filtration technology for removing water borne pathogens and reducing turbidity. The sand capable improving the physical, chemical, and microbiological quality of water in a single treatment process without the addition of chemicals and can produce an effluent low in turbidity and free of bacteria, parasites and viruses.
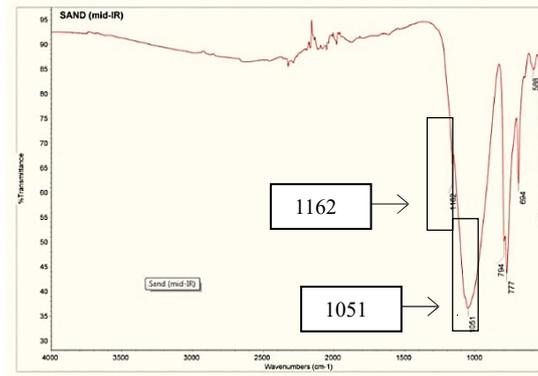
Fig. 3 - FTIR analysis result of sand

Table 7 - The FTIR spectral characteristics of sand

Wavelength Range (cm^{-1})	Wavenumber (cm^{-1})	Identified group	Reference
1300 - 1000	1162 1051	-C-O or –C-N bonds	[30]

3.3 Batch Experiment Analysis

3.3.1 Effect of pH

As it can be observed from Fig. 4 and Fig. 5 percentage of removal Ca^{2+} and Mg^{2+} increased proportional to pH value increment from 6 to 7. The maximum removal was observed at pH 7. Similar finding have been observed in other studies [33]. When pH increase over 7, the hydrolysis of metallic ions could take place which leading to decrease in biosorption process [28]. The decrease in percentage of removal above pH 7 for both metal was probably due to the precipitation by the formation Ca^{2+} and Mg^{2+} on the media surface.

3.4 Effect of Contact Time

3.4.1 Contact Time

The removal of Ca^{2+} and Mg^{2+} ions increases with time and attains saturation in about 60 minutes as in Fig. 6 and Fig. 7. The percentage of removal Ca^{2+} and Mg^{2+} ions amounted 89% and 84% respectively at equilibrium time of 60 minutes. An increment in contact time leads to an increase in metal removal but beyond a certain value of contact time, the percentage removal drops. The rate of metal removal is higher in the beginning due to large surface area of biosorbent being available for the adsorption of the metals [25].
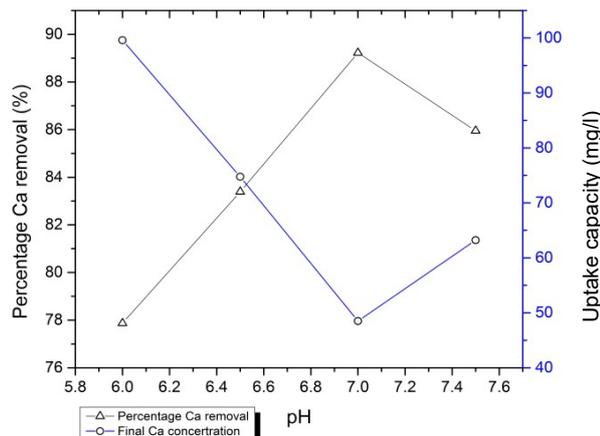

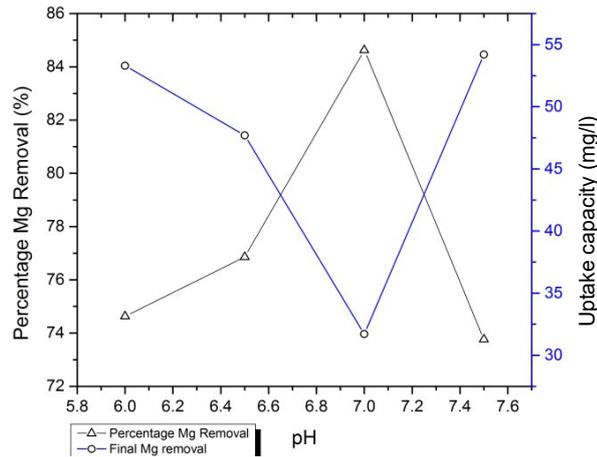**Fig. 4 - Effect of pH on the biosorption of Ca2+ onto sand, zeolite and RHAC**

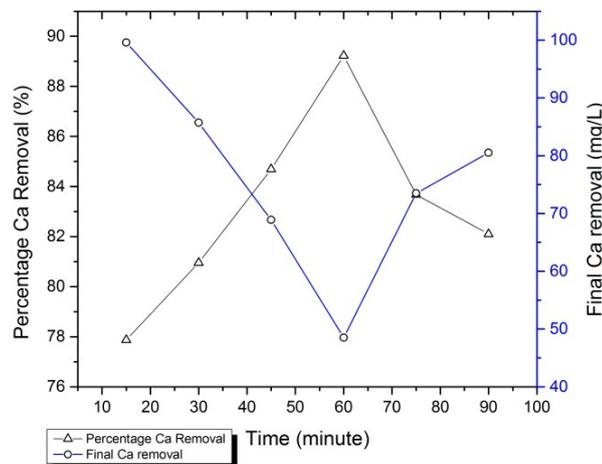Fig. 5 - Effect of pH on the biosorption of Mg^{2+} onto sand, zeolite and RHAC

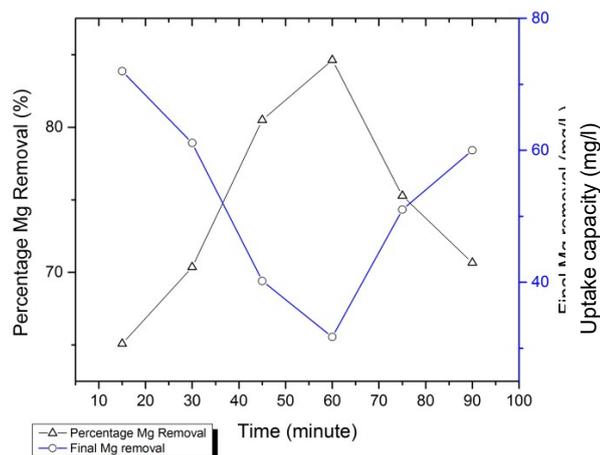Fig. 6 - Effect of contact time on biosorption of Ca^{2+} onto sand, zeolite and RHAC

Fig. 7 - Effect of contact time on biosorption of Mg^{2+} onto sand, zeolite and RHAC

The results obtained shows that sorption process can be divided in two stages. The first stage is rapid and quantitatively predominant while the second stage is slower and quantitatively insignificant. The rapid stage is probably due to the abundant availability of binding sites on the biosorbent, while in later stage, gradual occupancy of the binding sites cause diminution in sorption capacity. This is in agreement with similar finding reported by other researchers [34].

3.5 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) image for every media before and after treatment, it will extensively use to characterize the microstructure of every media surface. After treatment image after the media reach the equilibrium condition. The change on surface condition can be seen clearly where after the treatment all the media surface will show the appearance of new substance on the surface. As in Fig. 8 to Fig. 13. Fig. 8 shows that zeolite is mostly flat structural surface, with fragmented arrangement of pieces. Meanwhile, Fig. 9 indicates the solid substance is clearly attached to zeolite surface.
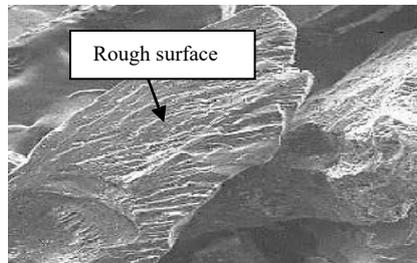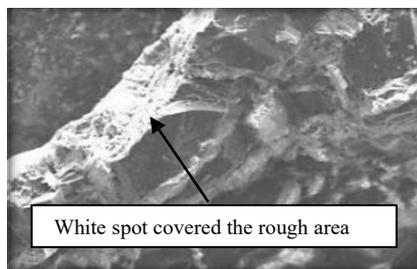

Fig. 8 - Zeolite before treatment

Fig. 9 - Zeolite after treatment

Fig. 10 shows pores and rough surface on RHAC before the treatment and Fig. 11 indicates rupture pores due to continuous metal loading on the RHAC surface.
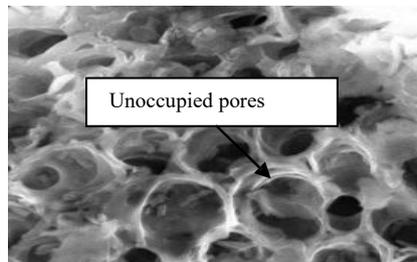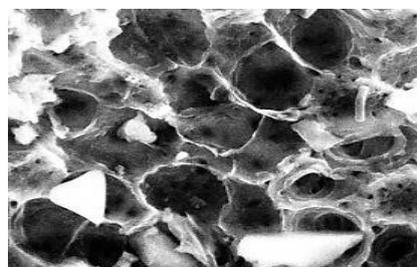

Fig. 10 - RHAC before treatment

Fig. 11 – RHAC after treatment

Fig. 12 indicate the rough surface texture; it will trap surface the suspended or dissolved solids. For Fig 13, the surface was covered with muddy or powdered substance, and the initial rock structure was covered. This situation

indicates that the sand trapped suspended solid substances onto surface, which is the main contribution of turbidity for groundwater.
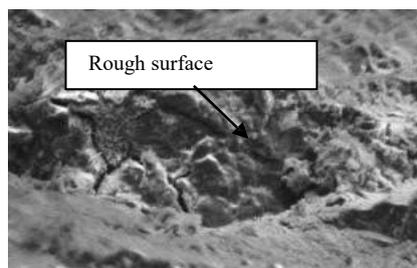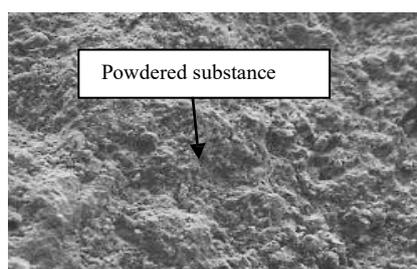

Fig. 12 – Sand before treatment

Fig. 13 – Sand after treatment

4. Conclusions

Sand, zeolite and RHAC are potentially to be developed into low-cost and environmentally friendly biosorbent for effective treatment of metal in groundwater. pH is one of the most crucial environmental variables governing the biosorption of the metal ions by biosorbent. It is not only influencing the functional groups but also the solution chemistry of the heavy metals. Contact time is also significant factor that effects metal adsorption. It was found that the greater amount adsorption as the contact time increased.

Acknowledgement

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