Electronic Journal of University of Aden for Basic and Applied Sciences
EJUA-BA Vol. 3 No. 4 (2022)
https://doi.org/10.47372/ejua-ba.2022.4.196
ISSN: 2708-0684

RESEARCH ARTICLE

A STUDY ON THE DEFLUORIDATION FROM WATER BY USING LOCAL LIMESTONE

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Received: 30 September 2022 / Accepted: 07 December 2022 / Published online: 31 December 2022

Abstract

Removal of excess fluoride [F-] from the water has been attempted by several authors by using different materials both natural and artificial. The main aim of this study was to the fluoride removal by using the local limestone adopting column method. The use of limestone obtained from the National Company of Cement (NCC) at Wadi Saim deposit and Wadi Nakhleen deposit, in Lahj Governorate, Yemen. X-ray spectroscopy analyses revealed that that limestone are classified as high and very high pure limestone, respectively. Local limestone has fluoride removal abilities by phosphoric acid (PA)- local crushed Limestone treatment (PACLT), with the treated water conforming to WHO guidelines for Fluoride in drinking water. Fluoride removal and pH of treated water attained stability within 3*h* for defluoridation from 10 mg/L [F-], to 0.20 mg/L with 0.68 mM [PA]. The results may be useful for suitability of local limestone for fluoride removal from drinking water.

Keywords: Fluoride removal of water, Local limestone, Phosphoric acid, Lahj, Yemen.

1. Introduction

Fluoride contamination of groundwater is a serious problem in several countries spread throughout the world as ingestion of excess fluoride, most commonly, through drinking contaminated groundwater causes fluorosis. Water pollution has become an inevitable and yet serious problem for human consumption. Fluoride exists fairly abundantly in the earth's crust and can enter groundwater by natural processes. Fluoride in drinking water has an intense effect on teeth and bones. Up to low level Fluoride concentration 1–1.5 mg/l, this gives strengthens the enamel. Concentrations of Fluoride in the range of 1.5–4 mg/l result in dental fluorosis whereas with prolonged revelation at still higher fluoride concentrations 4–10 mg/l result dental fluorosis progresses to skeletal fluorosis. Concentrations of high fluoride in groundwater more than 30 mg/l, occur widely, in many parts of the world [1, 2]. Examining the concentration of fluoride in different areas might help to ascertain proper preventive measures. Due to the effects of fluoride on human health, Fluoride concentration level in water supplies have been the subject of various recent studies [3]. Excess of fluoride in groundwater has become a threat in recent days due to the lesser availability of potable groundwater resource.

High fluoride concentrations in groundwater, mainly due to the geology strata (mineral composition) and physical properties (soil textures) [4, 5, 6]. Among these methods, adsorption is a widely used method for defluoridation of water because of its easy method of operation and cost effectiveness. Some of the adsorption materials broadly used for defluoridation are; physico-chemically treated sand [7], microwave assisted activated carbon [8], aluminum sulfate treatment [9], pumice [10] and raw Bauxite [11]. An extensive survey on the removal of excess fluoride in water shows that different techniques has been attempted by several authors, using natural and synthetic material. However, a fluoride removal method which is low-cost, safe, environment-friendly and can be used by a layman without requiring power is still not available. Another defluoridation method that has been reported is the PA-crushed limestone treatment (PACLT) technique which uses limestone and phosphoric acid to treat excessive fluoride. This method has shown to have very effective results, treating fluoride concentrations from 0.526 mM to 0.5-52.6 μM with final neutral pH in treated waters [12].

During using limestone and phosphoric acid have precipitation of CaF_2 and adsorption on limestone occurring. Below are the equations describing the mechanisms in the PACLT method [12]:

$$CaCO_{3(s)} + 2H_3PO_4 \rightarrow Ca^{2+} + 2H_2PO_4^- + CO_2 + H_2O \quad \text{Eq. (1)}$$

$$CaCO_{3(s)} + 2H_2PO_4^- \rightarrow Ca^{2+} + 2HPO_4^{2-} + CO_2 + H_2O \quad \text{Eq. (2)}$$

$$Ca^{2+} + F^- \rightarrow CaF_{2(s)} \downarrow \quad \text{Eq. (3)}$$

$$5Ca^{2+} + 3HPO_4^{2-} + 4OH^- \rightarrow Ca_5(PO_4)_3OH_{(s)} \downarrow + 3H_2O \quad \text{Eq. (4)}$$

$$Ca_5(PO_4)_3OH_{(s)} + F^- \rightarrow Ca_5(PO_4)_3F_{(s)} + OH^- \quad \text{Eq. (5)}$$

The dissolution of $CaCO_3$ by phosphoric acid, "the precipitation of CaF_2 and the precipitation of calcium phosphate hydroxide are completed rapidly" (Eq. 1, Eq. 3, Eq. 4) [12]. The sorption of fluoride with hydroxyapatite ($Ca_5(PO_4)_3OH$) is completed in about 3 hours (Eq. 5) and the neutralization of pH takes over 24 hours. The pH of the acidified water was 1.6 using 100 mM of PA [12]. This PACLT method was applied in a field trial in Assam, India for a small community and for 5 households. The community unit had a capacity to treat 220 L of water, while the household units had a capacity to treat 15 L of water at a time. They treatment units have treated water consistently up to 250 cycles. The PACLT treatment system could remove fluoride concentrations down to 0.001 mg/L [13].

The importance of the study is in the presence of a high fluorine in groundwater as well as drinking water. The presence of fluorine in groundwater is a natural sources due to the geological installation in those areas such as the Al-Muasymeer district, and hot springs water in Shara'a and Kirsh for Lahj Governorate [14], as well as in some Governorates in Yemen such as Al-Dhalea [5, 15, 16], Taiz [17, 18], Ghail Bawazeer district (Hadhramaut) [19], Dhamar [20], and Sana'a [21]. This technique has been applied to remove fluorine from drinking water in the Al-Muasymeer district, and the results will be published in another scientific paper later.We using local limestone and phosphoric acid to Defluoridation from Water.

The present study aimed to Fluoride Removal from Water by using local crushed limestone and phosphoric acid.

2. Materials and Methods

2.1. Study Areas

The study area is located at Lahej Governorate, Yemen. It is extending between Ar-Raqah Wadi Nakhleen at the west and Wadi Mahaar to the east, about 110 km to north from Aden. In UTM, international geographical position system is located between the coordinates 49°78'50" to 51°00'00" E and 14°70000 to 14°75'00" N [22].

The National Cement Company is located in Lahj Governorate, Yemen, about 68 km to north from Aden. Located between the coordinates 13° 20' North and 40°45' East.

Limestones were collected from different locations / mines of two sites of Yemen to conduct the experiments.The use of limestone obtained from the National Company of Cement (NCC), location at Wadi Saim deposit (Q-N.C.C.) and Wadi Nakhleen deposit (Q-W.N), in Lahj Governorate, Yemen.

2.2. Determination of limestone properties

The X-ray diffraction (XRD) analysis indicates the crude limestones to be high purity calcite baring from small amount of impurities as indicates by Energy Dispersive X-ray (EDX) analysis. The crude limestones were crushed with chips size between 0.5-1 cm using standard sieves.

2.3. Perperation of aquas solution (10 mg/L F^-)

Grade sodium fluoride (NaF) and PA were obtained from Marck and Midas Applied Materials Corp Origin Taiwan and used as such Fluoride stock solution of 1,000 mg/L concentration was prepared by adding NaF in DI water. Using this stock solution fluoride working solutions of 10 mg/L concentration were prepared. DI water was used for all the experiments.

2.4. Analysis of fluoride and pH

The concentration of Fluoride were measured by Using the Photometer, for the analysis 10 ml test tubes were filled with the water sample and the palintest photometer 600, Fluoride was analyzed calorimetrically using SPADNS as fluoride reagent solution, after their solution was left to stand for the specified time to allow for full color development, determination of the concentrations of Fluoride; reading was taken directly from the photometer (Figure 1). The pH was measured on Milwaukee pH meter, waterproof.

2.5. Defluoridation using limestone and phosphoric Acid

Batch tests were carried out with local crushed limestone using synthetic fluoride solutions with of 0.68 mM PA. In the first batch tests with local crushed limestone, a set of liter size bottles as column were filled with 2-3 mm size limestone particles and 10 mg/L F^- solutions has been used for the F- removal experiment with 0.68 mM PA, were added to the top level limestone (Figure 1). They were allowed to stand without shaking. In this experiment the treated water has been collected after different residence times from 1 hour of residence time up to 12 hours to observe the remaining F^- concentration and pH as a function of residence time. The experiment was carried out by according to the scientific method mentioned in the reference [23].

Pic (1): Palintest photometer 600

Pic (2): The limestone column

Pic (3): Phosphoric Acid 85%

Pic (4): SPADNS Reagent solution

Fig. (1): Instrumental Analysis, chemical and limestone column

3. Results and Discussion

3.1. Results of chemical analysis of limestone samples

Unsystematic distribution method of sampling from outcrop surfaces was carried out. Some small geological sections, fault zones and Wadi cuttings was used in sample collection; sample size ranged from 4 – 5 kg in weight. All samples are crushed, grinded, and pulverized to 0.07 mm grain size, 200 gm from each sample was taken for chemical analysis. All samples were analyzed in the laboratories National Cement Company, Quality Control Department, Lahj Governorate. The chemical analysis of limestones includes: CaO, MgO, SiO_2, Al_2O_3, Fe_2O_3, Na_2O, K_2O, SO_3, and LOI (Table 1).

Table (1): Results of chemical analysis of limestone samples from quarry limestone (QNCC) and Wadi Nakhaleen (QWN), Lahj Governorate, Yemen

NO.	Sample No	% OXIDE										
		SiO2	AL2O3	Fe2O3	MnO	CaO	MgO	SO3	Na2O	K2O	L.O.I	SUM
1	Q-W.N-1	0.56	0.13	0.089	-	55.92	0.39	0.017	0.019	0.017	42.59	99.732
2	Q-W.N-2	1.23	0.201	0.11	-	54.73	0.53	0.031	0.031	0.068	42.26	99.191
Mean		0.895	0.1655	0.0995		55.325	0.46	0.024	0.025	0.0425	42.425	99.4615
3	Q-N.C.C-1	1.66	0.21	0.36	-	49.86	2.16	0.11	0.044	0.079	41.2452	95.7282
4	Q-N.C.C-2	2.65	0.87	0.48	-	50.98	0.69	0.24	0.048	0.26	40.5165	96.7345
5	Q-N.C.C-3	2	0.13	0.12	-	54.16	0.48	0.027	0.036	0.052	42.768	99.773
6	Q-N.C.C-4	3.35	0.1	0.19	-	52.84	0.45	0.013	0.03	0.045	41.7057	98.7237
Mean		2.415	0.3275	0.2875		51.96	0.945	0.0975	0.0395	0.109	41.55	97.74

The samples of Wadi Nakhaleen limestone, two samples analyzed having the following, and are denoted by Q-W.N. The contents of SiO_2 from 0.56 -1.23% (mean 0.895%), CaO 54.73-55.92% (mean55.325%), MgO 0.39-0.53% (0.46%), K_2O 0.017-0.068% (mean 0.0425%), Na_2O 0.019- 0.031% (mean 0. 0.025%), SO_3 0.017-0.031% (mean 0.024%), AL_2O_3 0.13-0.201% (mean 0.1655%) and Fe_2O_3 0.089-0.11% (mean 0.0995%). These results are similar to the results of the study obtained by Ahmed *et al*., and Al Yamani [22, 24].

The samples of Quarry limestone of National Cement Company, four samples analyzed having the following, and are denoted by Q-N.C.C. The content of SiO_2 from 1.66 -3.35% (mean 2.415%), CaO 49.86-54.16% (mean 51.96%), MgO 0.45- 2.16% 0.945%), K_2O 0.045-0.26% (mean 0.109%), Na_2O 0.03- 0.048% (mean 0.0395%), SO_3 0.013-0.24% (mean 0.0975%), AL_2O_3 0.1-0.87% (mean 0.3275%) and Fe_2O_3 0.12-0.48% (mean 0.2875%).

As a result of chemical analysis, it is obvious that the highest content of CaO amounted to 55.92% is found within the extent of Wadi Nakhaleen, Al-Malah Directorate, with 54.73-55.92% distributed in both areas. The limestones could be classified as very high to medium purity of calcium carbonate.

3.2. Defluoridation by limestone and phosphoric Acid

The results of batch experiments of F^- removal with local crushed limestone chips of 3-4 mm size with PA, the remaining F^- concentration and pH as a function of residence time, are shown in Fig.2. It was presumed that limestone starts react with PA forming calcium phosphate and HAP as soon as PA containing F^- water added to the column Fluoride should be precipitated by limestone as CaF_2 as well as adsorbed simultaneously by limestone and HAP. Therefore, it was expected that the removal of Fluoride may start immediately after the addition of the water to the limestone column. In this experiment, the treated water has been collected after different residence times from 1 hour of residence time up to 12 hours to observe the remaining F^- concentration and pH as a function of residence time.

Figure (2) and Table (2) illustrates the percent removal of the Fluoride from initial 10 mg/L in presence of 0.68 mM PA using 2-3 mm size of limestone for 1*h*, 2*h*, 3*h*, 6*h* and 12*h* of residence time. In Table (2)and Figure (2) the remaining fluoride concentrations of the treated water along with pH after 1, 2, 3, 6 and 12 h of treatment time have been presented. It be clearly observed from Figure (2) and Table (2) that there is a little difference between the remaining F^- concentrations after 3,6 and 12 h, as was observed in Figure (3), the remaining Fluoride with n = 1 were found to be 1.12 mg/L (at 3 h and pH 6.53) and 0.73 mg/L (at 12 h and pH 7.03).

Table (2): The amount of remaining fluoride and pH of the treated water starting with initial 10 mg/L F^- containing 0.68mM Phosphoric Acid before filtration through limestone column after different time intervals.

N	[F-] - 10 mg/L					pH - 2.45				
	1 *h*	2 *h*	3 *h*	6 *h*	12*h*	1 *h*	2 *h*	3 *h*	6 *h*	12*h*
1	1.98	1.27	1.12	0.94	0.73	5.96	5.74	6.83	6.94	7.03
2	0.90	0.77	0.59	0.47	0.35	5.65	6.03	7.14	6.93	7.08
3	0.87	0.72	0.37	0.31	0.28	6.14	6.23	7.12	7.23	7.47
4	0.79	0.64	0.31	0.86	0.34	6.60	6.71	7.23	7.31	7.51
5	0.52	0.58	0.28	0.73	0.61	6.96	6.74	6.79	6.74	7.78
6	0.68	0.51	0.20	0.82	0.79	6.65	6.03	6.43	6.70	6.64
7	0.77	0.81	0.46	0.64	0.84	7.4	7.11	7.03	7.12	6.58
8	1.03	1.04	0.58	0.71	0.91	7.18	7.20	7.09	7.23	6.44
9	1.08	1.02	0.94	0.98	0.87	7.20	7.23	7.12	7.12	7.25
10	0.93	0.81	0.82	0.93	0.69	7.02	7.16	7.28	7.33	7.42
11	1.02	0.84	0.78	1.01	0.58	7.17	7.24	7.30	7.33	7.38
12	0.86	0.91	0.72	0.94	0.73	7.4	7.15	7.22	6.89	7.42
13	0.99	0.86	0.81	0.67	0.59	6.22	7.22	7.32	7.41	7.59
14	0.97	0.90	0.75	0.87	0.78	7.08	7.14	7.23	7.38	7.45
15	0.74	0.79	0.62	0.76	0.64	7.06	7.11	7.22	7.28	7.32

Estimation of the limestone for defluoridation of water using 0.68mM PA has been estimated by considering the different experimental parameters. The results of this estimation have been presented in the table (2) which show high capacity and hence efficiency of the treatment process.

Both experimental are expected to bring down fluoride from 10 ppm to 0.20 ppm at 3 *h* with 0.68 mM dose of PA. The final pH is expected to settle between 6.61 at 3*h* and 7.53 (the acceptable range being 6.5 to 8.5.

The fluoride removal performance of the local limestone in presence of phosphoric acid is impressive; however, neutralization of phosphoric acid did not required more time than the residence time used in these experiments viz, 1,2,3,9 and 12 *h* (Fig. 2). The water after treatment does not need pH correction. The carbonate of the limestone will neutralize the water. It has been observed that in phosphoric acid experiment with [PA] = O.68Mm at 3*h* pH is around 7.09, Even after 12 *h* of residence time the pH of the effluent water remains around 7.25, However, the remaining fluoride concentration is similar after 3*h* and 12*h* (Table 2).

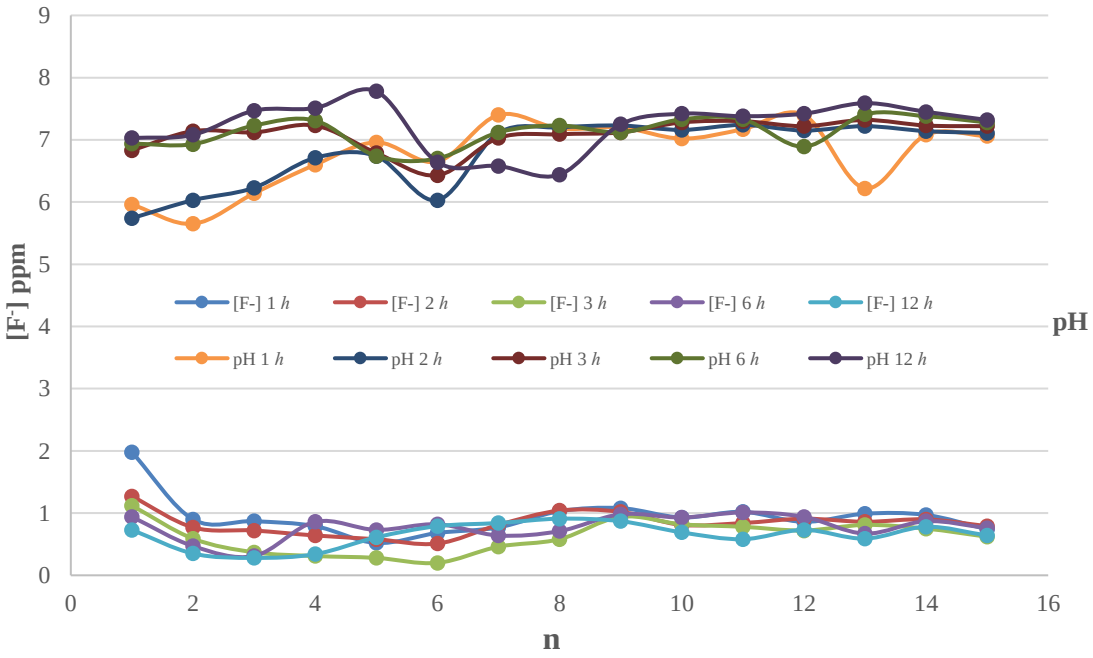

Fig. (2): the present removal of fluoride at initial concentration of fluoride 10 mg/L: after 1*h*, 2*h*, 3*h*, 6*h* and 12*h*, using 0.68 mM phosphoric acid. (The plot of present [F^-] removal (primary Y axis) and pH (secondary Y axis) vs. number of repeated use of the same limestone (n) after addition of 0.68mM phosphoric acid.

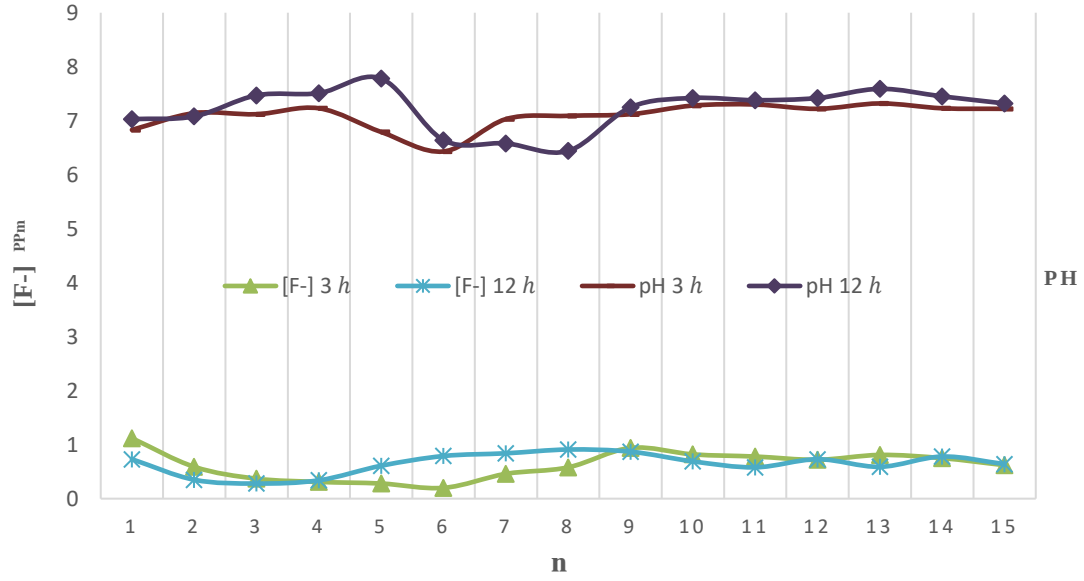

Fig. (3): the plot of present [F^-] removal (primary Y axis) and pH (secondary Y axis) vs. number of repeated use of the same limestone (n) after addition of 0.68mM phosphoric acid to the initial 10 mg/L fluoride water before treatment using the limestone column for 3*h* and 12*h*.

4. Conclusions

1. In this experiments removal fluoride from water using local limestone with PA. fluoride removal has been achieved to below 1 mg/L from an initial 10 mg/L within 2*h* of treatment time with 0.68mM of Phosphoric acid.
2. The removal fluoride capacity has been increased with increase in treatment time. Most of the [F-] was removed from the water within local limestone and after that the removal takes place slowly. There is a very little increase in the fluoride removal after the treatment time of 3*h* up to 12*h*.
3. The method removal fluoride using local limestone with phosphoric acid is safe and has been applied in other country.
4. These results of the study may help in selecting a suitable local limestone sample for using in removal fluoride from drinking water by phosphoric acid (PA)-local crushed Limestone treatment.

5. Recommendations

The study recommends applying this technique in removing high fluoride from groundwater and drinking water. This technique is easy to apply and less expensive (low cost) due to availability of raw materials and safe (non-hazardous).

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مقالة بحثية

دراسة حول إزالة الفلور من المياه باستخدام الحجر الجيري المحلي

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استلم في: 30 سبتمبر 2022 / قبل في: 07 ديسمبر 2022 / نشر في 31 ديسمبر 2022

المُلخّص

حاول العديد من الباحثين إزالة الفلوريد الزائد من المياه باستخدام مواد مختلفة طبيعية وصناعية. الهدف الرئيسي من هذه الدراسة هو إزالة الفلورايد من المحلول المائي باستخدام الحجر الجيري المحلي. استخدم الحجر الجيري الذي تم الحصول عليه من الشركة الوطنية للأسمنت (NCC) من رواسب وادي صاعم والموقع الآخر من وادي نخلين بمحافظة لحج، اليمن. بينت نتائج التحليل الطيفي بالأشعة السينية أن الحجر الجيري يصنف حسب درجة نقاوته الى حجر جيري عالي جداً وهذا بنطبق على رواسب وادي نخلين وعالي بالنسبة لموقع وادي صاعم. يتمتع الحجر الجيري المحلي بقدرات في إزالة الفلورايد عن طريق إضافة حمض الفوسفوريك (PA) مع الحجر الجيري المسحوق المعالج (PACLT)، أثبتت نتائج الدراسة توافق مواصفات المياه بعد المعالجة مع الحد المسموح به لتركيز الفلوريد في مياه الشرب وفقا لمعايير منظمة الصحة العالمية. حققت نتائج إزالة الفلوريد ودرجة حموضة من المياه ثباتًا خلال 3 ساعات لإزالة الفلور من 10 مجم / لتر [F-] إلى 0.20 مجم / لتر مع 0.68 ملي مولار حمض الفوسفوريك [PA]. ممكن الاستفادة من نتائج هذه الدراسة في استخدام الحجر الجيري المحلي لإزالة الفلوريد الزائد من مياه الشرب.

الكلمات المفتاحية: إزالة الفلورايد من المياه، الحجر الجيري المحلي، حمض الفوسفوريك، لحج، اليمن.

How to cite this article:

Sh. M. K. Saleh, R. M. Saleh and A. Hasen, “A STUDY ON THE DEFLUORIDATION FROM WATER BY USING LOCAL LIMESTONE”, *Electron. J. Univ. Aden Basic Appl. Sci.*, vol. 3, no. 4, pp. 276-283, Dec. 2022. DOI: https://doi.org/10.47372/ejua-ba.2022.4.196