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Activated carbon (produced from NaOH-pretreated palm trees base frond) as removal for Hydrogen Sulphide and Hardness from groundwater.

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Abstract

The study involved the ability of activated carbon prepared from palm trees base frond which is available in plenty amounts to adsorption hydrogen sulphide gas and hardness from groundwater. The palm trees base frond was activated by chemical methods using NaOH in the ratio of 1:5 NaOH with carbon . The characteristics of activated carbon and its FTIR spectrophotometry was studied.A treatment was performed on water well of high concentration hydrogen sulphide and hardness at three pH (5,7,9). The removal of sulphide ion was high efficiency of 97.7% at pH5.Also , the result showed the best removal of hardness was achieved at pH9 with efficiency of 45.2% .The fluoride was completely removed at pH7 in addition to good removal of SO42- at pH5 and silica at pH7. An increase in sodium ion concentration was found because of treatment with NaOH.

Key words: Activated carbon, NaOH, palm trees base frond, Hydrogen Sulphide.

الكاربون المنشط المحضر من كرب النخيل باستخدام هيدروكسيد الصوديوم لازالة كبريتيد الهيدروجين والعسرة من المياه الجوفية

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خلاصة

تضمنت الدراسة قدرة الكاربون المنشط المحضر من كرب شجر النخيل المتوفر بكثرة لامتزاز غاز كبريتيد الهيدروجين وعسرة المياه من المياه الجوفية. تم تنشيط كرب النخيل بالطريقة الكيميائية باستخدام هيدروكسيد الصوديوم وبنسبة 5:1 هيدروكسيد الصوديوم الى الكاربون. درست خصائص الكاربون المنشط وكذلك طيف الاشعة تحت الحمراء. اجريت المعالجة على مياه بئر ذو تركيز عالي من غاز كبريتيد الهيدروجين والعسرة باستخدام ثلاث مديات من الدالة الحامضية . (5,7,9) pH اعطت المعالجة كفاءة ازالة 97.7٪ لأيون الكبريتيد عند 5 pH . كما بينت النتائج ان افضل ازالة للعسرة عند 9 pH وبكفاءة 45.2٪ . ازيل الفلوريد بصورة تامة عند 7 pH وكذلك ازالة جيدة لأيون الكبريتات عند 5 pH والسليكا عند 7 pH. لوحظ وجود زيادة في تركيز ايون الصوديوم لان المعالجة تمت باستخدام هيدروكسيد الصوديوم.

1. Introduction

Hydrogen sulphide is a flammable, colorless gas. Sulfur containing compounds in general (such as mercaptants) and Hydrogen sulphide in particular are the products of the biodegradation of sulfur containing amino acids such as cysteine and methionine by anaerobic bacteria. It is also a product of anaerobic reduction of sulfate (SO_4^{2-}) (1). In the aqueous phase $H_2S(g)$ is a diprotic weak acid (2) . Hydrogen sulfide exists naturally in many natural gas wells, in waste-water treatment plants(3).

The hardness in water is a result major of calcium and magnesium cations in water. The hardness in water is naturally arising in groundwater which ion exchange and weathering of sedimentary rock limestone, and calcium bearing minerals (4).formations in pipes and on distribution system (5). Therefore, it is necessary to find a method for removal of hardness in water supply for deference uses. Activated carbons (AC) are known as very effective adsorbents due to their highly developed porosity, large surface area (that can reach 3000 $m^2 g^{-1}$), variable characteristics of surface chemistry, and high degree of surface reactivity(6). These unique characteristics make AC very versatile materials, which have been studied not only as adsorbents, but also as catalysts and catalyst supports used for different purposes such as the removal of pollutants from gaseous or liquid phases and the purification or recovery of chemicals.Recent study showed used activated carbon removal of mercury from aqueous solution(7) , used jamun seed derived activated carbon by NaOH as an adsorbent for removal of fluoride from water(8),prepared activated carbon from palm trees base frond to removal of sulfide from the water sulfur spring with efficiency 99%(9) , used activated carbon prepared from banana peel (which consider as fruit)waste for the removal of total sulfide from spring water(10). However, due to their high production costs, these materials tend to be more expensive than other adsorbents(11). Chemical activation involves $ZnCl_2$, H_3PO_4, NaOH and KOH have been used for chemical activation (12).These chemical agents develop the porosity based on dehydration and degradation. Generally, chemical activation (300-500 ^{0}C) takes place at lower temperature than physical activation (13). In Chemical activation, both Carbonisation and activation may be performed in a single-stage process, by employing chemical activating agents such as $ZnCl_2$, KOH, NaOH (14). Therefore resulting in the development of a better porous structure, although the environmental concerns of using chemical agents for activation could be developed (15). High surface area activated carbons were prepared by simple thermo-chemical activation of Jatropha curcas fruit shell with NaOH as a chemical activating agent. Sodium hydroxide was also shown to be more particularly interesting because NaOH activation can reduce chemical activation cost and environmental load when compared with KOH activation (16). One of the most important advantages of chemical activation over physical activation is the lower treatment temperature and shorter treatment time. In addition, the activated carbon obtained by the chemical activation possesses larger surface area and well controlled microporosity in smaller ranges (17). The aim of this study is to treat the groundwater of high H_2S and hardness concentration using the activated carbon prepared from palm trees base frond .

2. Materials and methods

2.1 Preparation of activated carbon :

The dry part of palm tree washed with water and cut into small pieces and washed by distilled water several times to removal dissolved salts and undesirable material. The pieces were dried at 120 ^{0}C in oven for 48 hours .The sample was burnt in muffle furnace at 350 ^{0}C for two hours . The size of produced carbon using coarse sieves selected (150 to 380 µm) specialized for this purpose . Equivalent quantities of NaOH and carbon , 1:5 were transferred in two a beaker and sufficient volume of distilled water was added and stirred with magnetic stirrer for 1 hour with a rate of 4 rpm in order to give NaOH the sufficient time to penetrate through the pores . The carbon was dried at 120 ^{0}C to removal H_2O ,and then put in muffle furnace at 340 ^{0}C for two hours . The produced carbon was washed several times to removal sodium ions and then dried in oven at 120 ^{0}C in order to use it in the different types of treatments(9) .

2.2 Methods of determinations of carbon characterstics of activated carbon characterstics measurements :

2.2.1 Density measurement:

A definite quantity of carbon was transferred into a graduated cylinder (size 10cm^3) and slightly in passed in order to remove the pores between carbon particles in order for the sample to contain its volume on condition that the carbon particles should be one level the mark of cylinder. The sample was weighed using sensitive balance and the density was calculated according to the following relation(18) .

Density (g/cm^3)= mass(g)/ volume (cm^3)…..(1)

2.2.2 Determination of Humidity Content

1 gram of carbon specimen was taken into weighed small beaker at 150 ^{0}C in an oven for 3 hours . The sample was transferred into desiccator and left for cooling . The beaker with its contents was weighed by sensitive balance. From weighing difference, the percentage of humidity in the specimen was calculate (19).

2.2.3 Determination of Ash

1 gram of activated carbon was weighed and transferred into weighed crucible in an oven at 100 ^{0}C for 1 hour .The crucible was left for cooling in a desiccator and weighed by sensitive times with all types of carbon and the percentage of ash was calculated in each specimen (20).

2.2.4 Evaluation of adsorption activity for prepared activated carbon speciments

a- Adsorption iodine from its aqueowous solution. Iodic number method is simple rapid and gives indication to the internal surface area for activated carbo .The iodic number is defined as the number of mg of iodine adsorbed from aqueous solution by 1gm of activated carbon .The weight of adsorbed iodine by carbon speciments is calculated by applying the following relation :

X= A- (2.2B× VOLUME thiosulphate ….(2)
A = N1 × 12693
B = N2 × 126.93
Where X = weight of iodine adsorbed by activated carbon solution
N1 = Normality of iodine (0.1N)
N2 = Normality of thiosulphate solution 0.1N

Iodic number was calculate from the following equation : I,N=X/M×D(21)

b- Adsorption of methylene blue from its aqueous solution .This type of adsorption is an indication to the surface area of the activated carbon and referred to the ability of activated carbon to adsorb the big molecules . Methylene blue value is defined as number mgs of methylene blue that removed from its aqueous solution using o.1 of activated carbon (22).

2.2.5 pH measurement.

10 ml of distilled water was added to 1gm of activated carbon in a beaker and well shaked by shaker instrument at 25 0C for 30 minutes .The solution was filtered and the pH of the filtrate was measured by pH meter .The process was repeated several times and the mean value was taken as a measure of pH (22).

2.2.6 FT-IR measurement.

The spectra of IR for activated carbon and non- activated carbon recorded by FT-IR spectrometer in a rigion of 400 - 4000cm^{-1} after mixing the speciment with pure solid potassium bromide (KBr) and modulated it as thin CD.

2.3 The optimum time and temperature for adsorption process

In order to get best adsorption , a constant weight , was taken from activated carbon samples. (1gm) with 50 ml of well J1water .The adsorption time was taken at (15,30, 60 ,120 minutes).After that , the time was taken constant at 30 min and the temp of adsorption as(10, 20, 30,40 ^{0}C) .(10gm) of activated and non- activated carbon with 500ml of well were taken for this process . It is concluded from adsorption was at 20 ^{0}C with 200rpm .

2.5 Water test(23)

1-5-1 Physical properties

Turbidity by turbidity meter .pH by pH meter. EC by conductivity meter.DO by dissolved oxygen meter .Temperature by-Thermometer.

1-5-2 Chemical properties

Total calcium and hardnesses were determined by titration methods with EDTA using Eriochrome black T and Murexide indicators. The chloride concentration was measured by titration with silver nitrate using potassium chromate as indicator. Alkanity is measured with diluted H_2SO_4 solution using methyl orange as indicator.Bicarbonate measurements were done with sodium hydroxide using phenolphthalein as indicator. Sulphate was determined by precipitation as $BaSO_4$ by addition $BaCl_2$ 10% in acidic solution . COD Ammonium ion determined by using kits . was determined using kits. Sulphide ion selective electrode combined electrode HANNA and fluoride was determined by ion selective electrode mettler Toledo.

3. Result and dissection

3.1 Activated Carbon Characterization

The product of activated carbon as mentioned in the past studies and researches depends on the primary materials which contain high content of carbon and activating it by physical and chemical methods which involves using acids or bases in order to increase the surface area for carbon .In our study the wood of palm trees stone frond was used for production of activated carbon . So , sodium hydroxide was used to activate carbon in the ratio of 5 carbon 1NaOH. The study involved also the comparison between carbon characterstics (activated). The study involved also applications the above both types of carbon(activated) in waters treatments .It is shown from the table (3-8) iodic number in case of using NaOH is more than of non activated carbon. This is due to role of NaOH in enlargic inner pores of carbon. In case of methylene blue , it was found an increase in its concentration in activated carbon NaOH has increased outer por sizes for activated carbon and then increasing adsorption .It was noted that the percent of ash from activated carbon was less which confirmed that the hydroxide solution and washing with distilled water has replaced large quantities of inorganic materials and ash components. The results of humidity in activated carbon is less .The reason is that the activated carbon has less ability to adsorb water , because of somewhat of large surface produced by activation when it dried .The density value of activated because the activating by NaOH increases the weight of carbon. other studies showed ,the use of chemical reagents in the activation process provides an increase in the amount of acid groups present in the ACs surface.

3.1.1 IR spectrum

The spectra of both activated and raw in their IR spectra which confirms appearance of new absorption bonds in activated carbon. Large bond at 3438cm-1due to OH^- group. Band at $1624cm^{-1}$ due to group (-C=C-). Two band at 1381 and 1433 cm^{-1} due to (C-H) group of raw carbon (fig3-1). The spectra of activated carbon showed large absorption band at 3437 cm^{-1} due to OH- group. New band at 1714 cm^{-1} due to C=O group. Sharp band at 1574 cm^{-1} due to(-C-C-). Sharp band at 1371 cm^{-1} due to(C-H) . Large and narrow bands at the frequency $1225cm^{-1}$ due to -(CH_2-X) group .Small band at 1112 cm^{-1} due to C-O group. (fig3-2). From the IR spectra of raw carbon and activated carbon It can be noted that there was disappearance of bands when comparing raw carbon spectrum with the activated carbon spectra, indicating that the chemical bonds were broken during the carbonization process followed by the activation.

3.2 Results of physical and chemical treatments on the waters of the well

The treatment using activated and unactivated carbon was completed at controlled temperature 20 ^{0}C. The result of groundwater gave lowered turbidity when activated were used .The percentage of turbidity was very little which does not exceed the permitted level after treatment .In case of treatment by activated carbon, the turbidity of all wells removal semi completed .The value of pH was highe when treatment with activated carbon because the activated carbon was active by NaOH .The results an illustrated decrease in EC values. The more at pH9, the reason for that was attributed to decrease in dissolved salts concentration .In case of TDS , the results showed decrease in dissolved salts was found when the groundwater was treated. The better efficiency was found at pH9 because of the decrease of cations and anions from water.The presence of activated carbon has lowered TSS to zero .In the case of dissolved oxygen uncertainties in oxygen levels for the dissolved oxygen in groundwater after treatment by activated carbon. The results of COD when used three treatment with variation pH showed low values of COD increasing pH, with large value at pH7 by treatment with activated carbon (75.4%) (24). It is noted that there are approaching in removal efficiency at pH7 and pH9 because of occurrence electrostatic attraction between organic compounds with positive charges and carbon surface which carries negative charges .In acidic pH , these compounds are decomposed and the existence of excess of H^+, there will be competition between H^+ and organic compounds carrying positive charges on the adsorption sites(25) .The results have given decrease in TH all treatments and all pH values. The removal efficiency increases with increasing pH and the best removal was at pH9, where the cations are adsorbed with OH^- groups produced in activation by activated carbon process (26,27) .The better removal was also at pH9 because of strong adsorption of cations (Ca^{2+} and Mg^{2+}) in the present of excess OH^- ions resulted from activation process .This indicate the removal of water hardness ions was strongly dependent on the pH of the solution. This is because variation of hydrogen ion concentration affects the number of metal ions binding sites on the adsorbent surface. That to say, at lower pH, the surface of the adsorbent is surrounded by hydrogen ions (H^+).The latter prevents adsorption of hardness ions on to the binding sites of adsorbent through repulsion(25). The better removal of Ca^{2+} and Mg^{2+} was also at pH9 because of strong adsorption of cations (Ca^{2+} and Mg^{2+}) in the present of excess OH^- ions resulted from activation process with efficiency of Ca^{2+} and Mg^{2+} (28) .An increase in sodium ions in case of using activated carbon , because the carbon was activated with sodium hydroxide . In case of K^+ ions, the best removal at pH9. The present of excess OH^- ions resulted from activation may adsorb the K ions. The best removal of fluoride ion is at pH5 .The pH of the solution is an important parameter in the adsorption process. The effective removal is observed in acidic and near neutral range. At lower pH ranges, the surface of the adsorbents could be positively charged, , which causes the strong interaction of fluoride with the adsorbents or partially positive charge of carbon due to the electrostatic interaction between positively charged adsorbents surface and negatively charged fluoride ions. But as the pH of the solution increased above 8, the number of OH^- groups are increased resulting in the decrease of positively charged sites(29,30).In case of chloride ion,the treatment results showed limited removal of all pH values and the larger was at pH9 values in the presence of activated carbon. In case of sulphate ion, the maximum SO_4^{2-} removal on activated carbon occurred at acidic pH at pH5. This can be due to the electrostatic attraction between the SO_4^{2-} anions and the positive charges located on the adsorbents surface. The effect of pH value showed the adsorption of sulfate is best efficiency removal at pH5. The decrease removal of SO_4^{2-} as a result of the rise in pH may be due to the fact that at higher pH, the adsorbents surface are negatively charged and subsequently enhances the electrostatic repulsion between sulphate ions and the adsorbents, leading to the release of the adsorbed species off (31,32) . The results show the best removal was at pH5 and pH7 while at pH9 hydroxyl ion also competed with $HS^-$and S^{2-} .During H_2S adsorption, besides physisorption onto the pore surface due to van de Waals forces, chemisorption by forming hydrogen bonding also occurred. Hydrogen atoms in H_2S could strongly interact with oxygen in form of hydroxyl (H-O) and carbonyl (C=O) and (C-C) groups due to the high electrostatic attraction. H_2S can therefore preferably adsorb onto active adsorption sites provided by oxygen functional groups via hydrogen bonds besides on pure carbon sites by formation of hydrogen bonds during chemisorption. In addition, due to the oxygen functional groups, H_2S was easily oxidised as follows(33).H_2S can react with the active sites on activated carbon by forming sulfur species or react with carbon atom directly(34).

$$C_nH_xO_y{+3}\ \ {+H_2S} \rightarrow C_nH_xO_y{+3}\text{....HSH.......}10$$

$$C_nH_xO_y{+3}\ \ {+H_2S} \rightarrow C_nH_xO_y{+3}\text{....HSH......}11$$

$$C_nH_xO_y{+3}\ \ {+H_2S} \rightarrow C_nH_xO_y{+3} + S\ \text{(element)} + H_2O\ \text{...}12$$

$$C + H_2S \rightarrow C\text{-}S\ ,C\text{-}SH\ \text{.......}\ 13$$

Conclusions

1- The treatment of carbon with NaOH has given large surface area of activated carbon .

2- The results of treatment with the activated carbon have given semi complete removal for sulphide ions because of the active sites of carbon which carry function groups and pores which adsorb sulphide ions .

3- The results of treatment have shown that the best removal of total hardness was at pH9 because of the precipitation of Ca^{2+} and Mg^{2+} ions in basic media ,in addition of OH^- groups which react with Ca^{2+} and Mg^{2+} at basic media .

4- Lowering in EC and TDS values after treatment as a result of decreasing the concentration of cations and anios. Decreasing of COD values which gave the best value at pH7 because of shortage of hydrogen ions which competes the organic compounds of positive charges on adsorption sites . Complete removal of fluoride ions .The best removal value of SO_4^{2-} was at pH5 and pH7 for silica removal. Increase of sodium ions as a result of carbon activation by NaOH.

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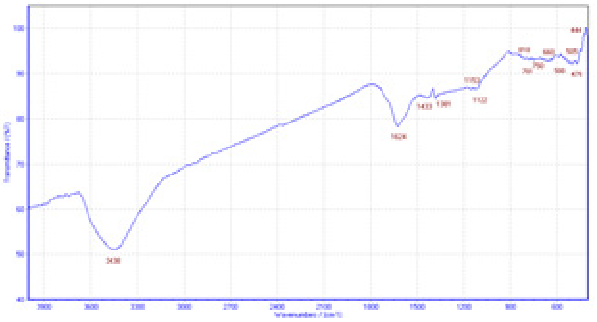
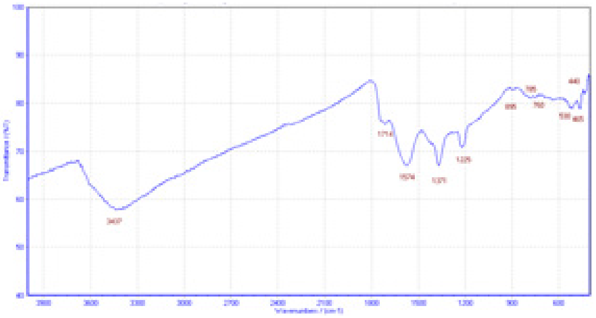
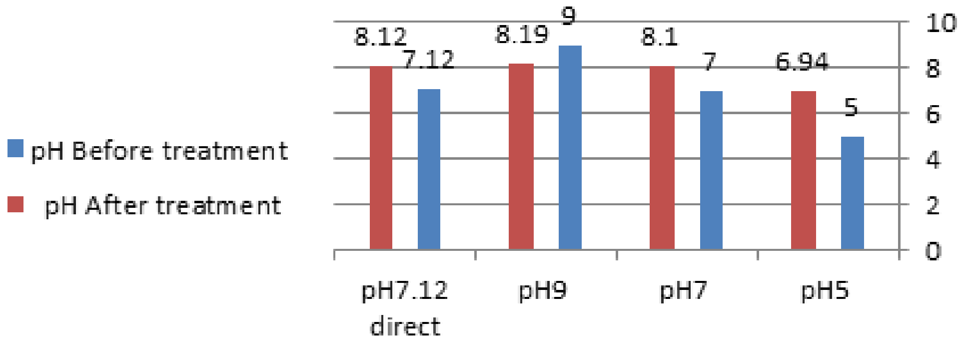


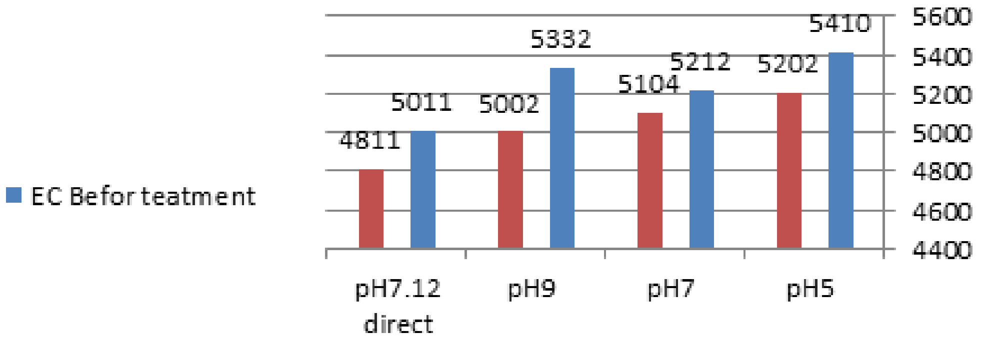
FT-IR spectra carbon before activation
Fig 3 - 1



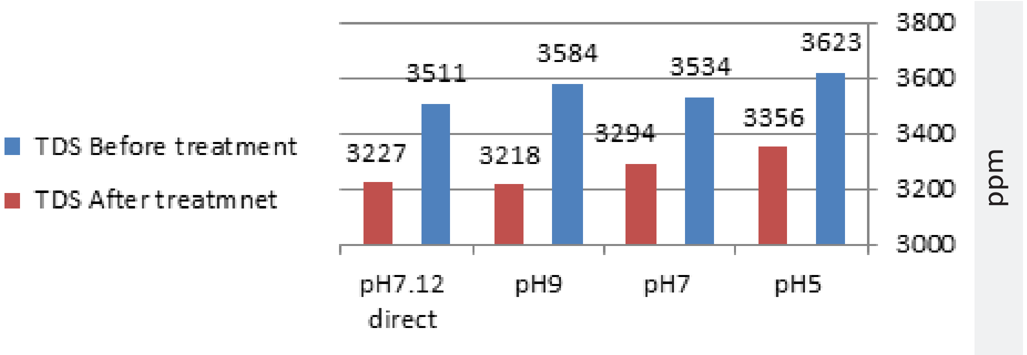
FT-IR spectra for activated carbon
Fig 3 - 2



Fig(3 -3)
pH Before and after treatment

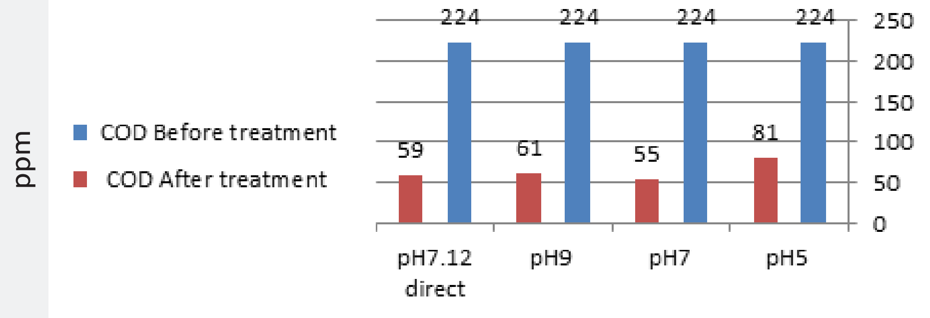


Fig(3 - 4)
EC Before and after treatment

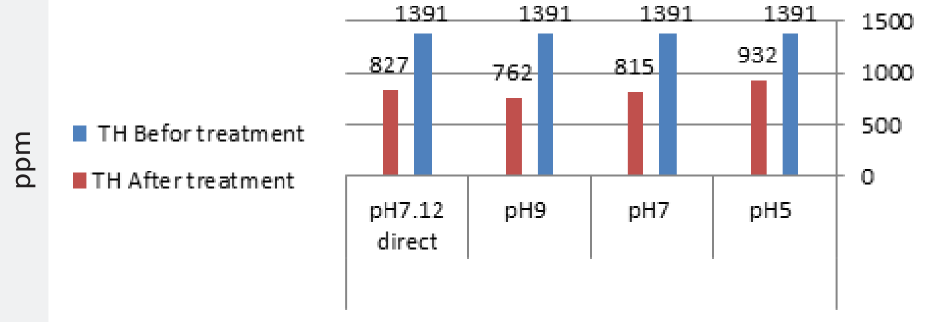


Fig(3 - 5)
TDS before and after treatment

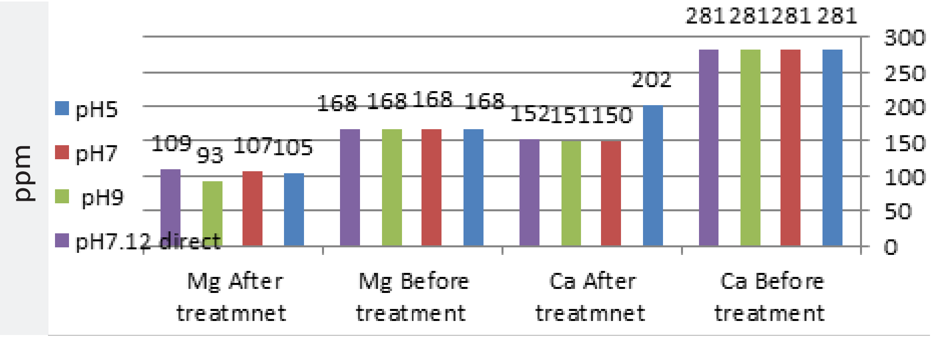
Fig (3 - 6)
COD before and after treatment



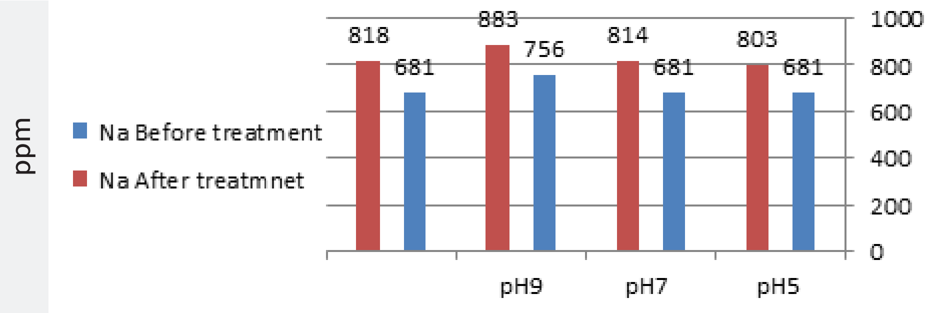
Fig(3 - 7)
TH before and after treatment



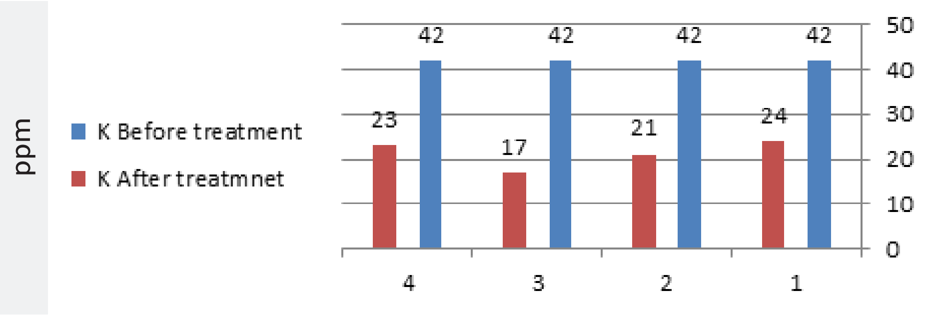
Fig(3 - 8)
Ca and Mg before and after treatment



Fig(3 - 9)
Na before and after treatment



Fig(3 - 10)
K before and after treatment



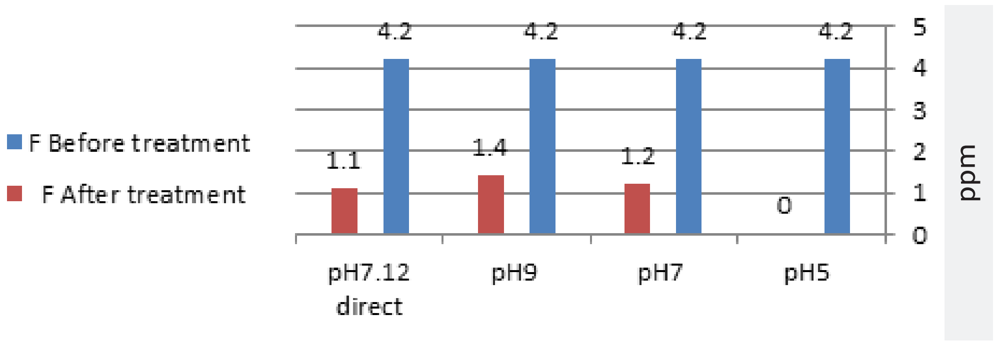


Fig (3 - 11)
F before
and after
treatment

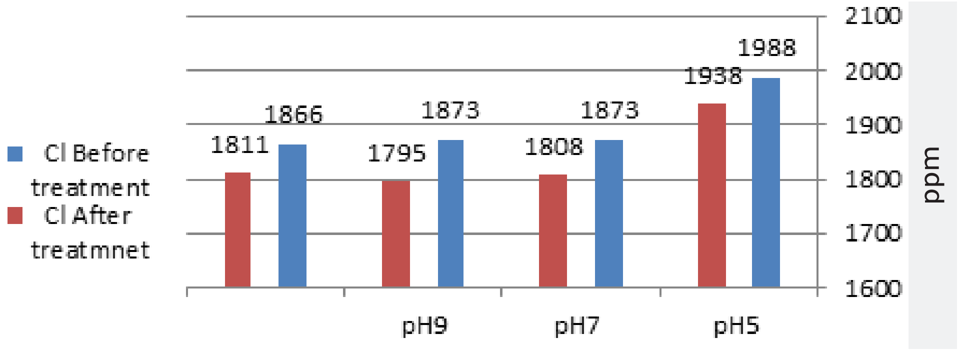


Fig (3 - 12)
Cl Before
and after
treatment

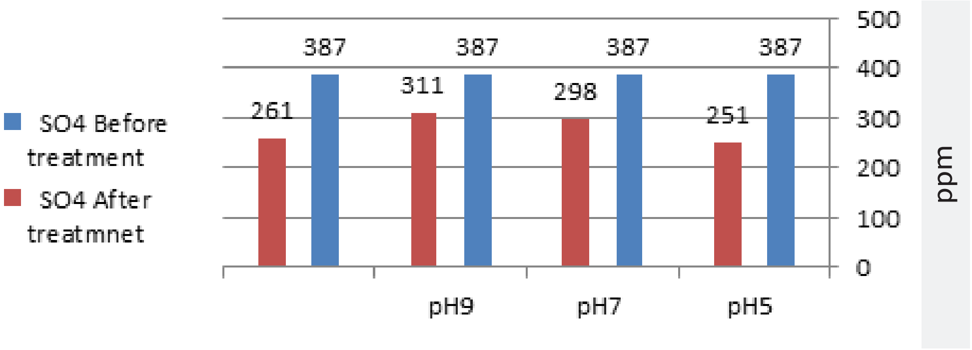


Fig (3 - 13)
Cl Before
and after
treatment

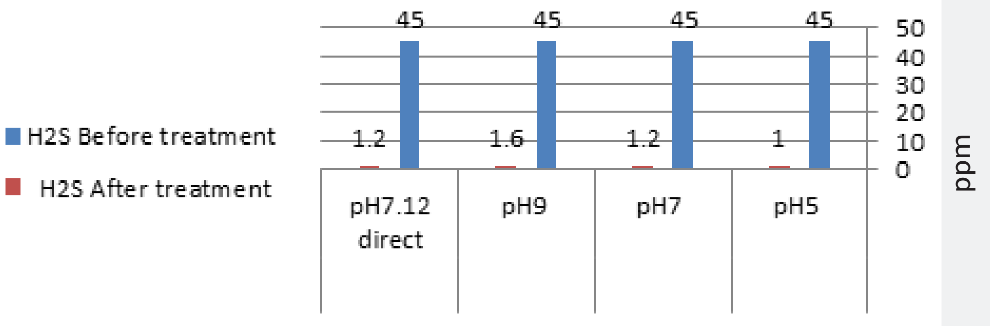


Fig (3 - 14)
H2S Before
and after
treatment

Table 3-1 Activated Carbon Characterization

Samples	Palm trees base frond:NaOH	Iodinenumber (mg/g)	Methyleneblue (mg/g)	Ash%	Humidity%	Density (g/cm3)	pH
AC	5:1	1016	51	0.73	0.767	0.31	8.68
BDH	-	908	90	3.2	0.8	3.2	-

Table 3-2 Efficiency of removal Sulphide ion by activated carbon

efficiency	ppm25	ppm50	ppm100	200 ppm	45ppmRaw water
pH5	98.5	96.7	93.41	89.5	97.7
pH7	98.1	96.1	92.3	86.2	97.3
pH9	97.3	95.2	90.4	83.1	96.4

Table 3-3 treatment of well water by activated carbon

Parameter	**Raw pH5**	**A.c**	**Raw pH7**	**A.C**	**Raw pH9**	**A.C**	**Raw**	**A.C**	**WHO standards (2011)**
Temp	**25**	**20**	**25**	**20**	**25**	**20**	**25**	**20**	
Turb	**3.6**	**0.1**	**3.6**	**0.2**	**3.6**	**0.2**	**3.6**	**0.1**	**5**
pH	**5.00**	**6.94**	**7.00**	**8.10**	**9.00**	**8.19**	**7.12**	**8.12**	**6.5- 8.5**
E.C	**5410**	**5203**	**5212**	**5104**	**5332**	**5002**	**5011**	**4811**	**500**
TDS	**3623**	**3356**	**3534**	**3294**	**3584**	**3218**	**3511**	**3227**	**500**
TSS	**6**	**0**	**6**	**0**	**6**	**0**	**6**	**0**	
D.O	**8.9**	**7.9**	**8.9**	**7.6**	**8.9**	**8.1**	**8.9**	**7.9**	**10**
COD	**224**	**81**	**224**	**55**	**224**	**61**	**224**	**59**	**10**
T.H	**1391**	**932**	**1391**	**815**	**1391**	**762**	**1391**	**827**	**100**
Ca^{2+}	**281**	**202**	**281**	**150**	**281**	**151**	**281**	**152**	**75**
Mg^{2+}	**168**	**105**	**168**	**107**	**168**	**93**	**168**	**109**	**50**
Na^{+}	**681**	**803**	**681**	**814**	**756**	**883**	**681**	**818**	**200**
K+	**42**	**24**	**42**	**21**	**42**	**17**	**42**	**23**	**10**
F^{-}	**4.2**	**BDL**	**4.2**	**0.9**	**4.2**	**1.4**	**4.2**	**1.2**	**1**
HCO_3^{-}	**98**	**42**	**71**	**3**	**0**	**0**	**68**	**7**	**500**
Alk	**18**	**21**	**153**	**121**	**234**	**52**	**155**	**112**	**250**
Cl^{-}	**1988**	**1938**	**1873**	**1808**	**1873**	**1795**	**1866**	**1811**	**250**
SO_4^{2-}	**387**	**251**	**387**	**298**	**387**	**311**	**387**	**261**	**250**
S^{2-}	**45**	**2**	**45**	**BDL**	**45**	**BDL**	**45**	**BDL**	**0.1**