

Extraction of sulphur from sulphur springs waters and the possibility of exploitation of these waters for human and agricultural purposes.



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ABSTRACT

The research involved preparation of local arrangement for sulphur extraction because of the danger of hydrogen sulphide on human health, its toxicity and environmental pollution. Iron (II) Chloride prepared from iron filings and HCl (1:1) was used to extract hydrogen sulphide as FeS which is useful as a source of H₂S and other sulphur compounds. The method offered high efficiency of extraction reached %99.09. Sulphide ion –selective electrode was successfully used to determine the sulphide concentrations before and after treatment which gave confidential results. Extraction with iron(II) Chloride was considered because of its availability, low cost and ease of reproducibility. Physical and chemical tests and determinations of springs waters showed very high levels of hardness and high levels of dissolved salts reached an average of 21656 ppm. Therefore, these springs waters even after treatment are not suitable for drinking, washing and agricultural purposes. High levels of heavy metals exceeded the permitted standard levels found in these springs waters have increased the danger of using these springs waters.

Introduction

Sulphur containing compounds such as mercaptans and H₂S are products of the biodegradation of sulphur containing amino acids such as cysteine and methionine by anaerobic bacteria⁽¹⁾. It is also a product of anaerobic reduction of sulphate by sulphate – reducing bacteria⁽²⁾. Combustion of a gaseous fuel which contains H₂S may lead to the formation of acid rain due to oxidation of H₂S to SO₂ which is furtherly oxidised to highly water soluble SO₃ forming corrosive H₂SO₄ which attacks different materials such as iron, copper, cement, pipelines, engines and biogas storage structures^(3,4). Exposure of human beings to low concentrations of H₂S can cause headaches, nausea and irritation of eyes and respiratory track as well as rhinitis and intense cough. High concentrations can cause paralysis of the breathing system, unconsciousness and finally death⁽⁵⁾. Sulphide is always present in ground water which is the main source of sulphur springs waters.

Several springs waters are distributed in Heat district (West of Ramaddi City) and the populations around these springs always complain from its undesirable smell and direct effects on their health^(6,7). The permissible H₂S concentration in clean water is in the range of 0.025 – 0.25 µg / L, while its range in springs waters is (210 – 422.4 ppm)⁽⁸⁾. Therefore it is important to adequately control H₂S for public health and safety as well as protecting the environment⁽⁹⁾. Many adverse technologies have been applied to reduce or remove sulphur which is H₂S from gaseous streams and aqueous solutions. The technologies involved oxidation of H₂S into sulphate and elemental sulphur which require high energy using Claus process⁽¹⁰⁾. Adsorption of H₂S on activated carbon, zeolite and iron oxide were used but it required frequent regeneration of the adsorbent as a result of saturation of the adsorbent which is high costly process^(10,11).

Adsorption of H₂S by highly alkaline solutions, chemical precipitation with FeCl₂ solution and oxidation by NaOCl, O₃ zone, KMnO₄ and hydrogen peroxide have relatively high efficiency in reduction and removal of H₂S⁽¹²⁾. All these processes require high reagent consumption and a proper final disposal of the spent solution in which the reagents can not be

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regenerated or reused ⁽¹²⁾. Another process was the regenerative gas scrubbing solvent that have high affinity to H₂S such as ethanol amines ⁽¹⁰⁾ but the process required high energy and operating costs ⁽⁴⁾. Liquid redox sulphur recovery (LRSR) process offered good hydrogen sulphide removal and highly flexible operating properties at ambient temperature ⁽¹³⁾, but the reactions tend to form undesirable sulphur oxyanions as byproducts ⁽¹⁴⁾.

The aim of this investigation was to apply unexpensive process for removal of H₂S from Heat springs waters and get advantageous of the products. Additionally, the water might be clean and free of H₂S and might be used after passing through filters, for human and agricultural purposes.

Experimenta part

Reagents and chemicals.

Common chemicals like Na₂S .5H₂O, NaOH, KI, AgNO₃, I₂, FeCl₂, FeCl₃, KNO₃, Na₂S₂O₃, KIO₃ anhydrous Na₂S, NaNO₂, and acids were of highly graded and purchased from Fluka and BDH companies. Double distilled water was used for all the solutions preparations Sulphide antioxidant buffer (SAOB)⁽⁵⁾; pproximately 300 mL of distilled water was taken to 500mL volumetric flask, 40gm of NaOH, 10gm of NaNO₂ and 35 gm of Na₂EDTA were added, swirled to dissolve completely and made up to 500 mL with distilled water and stored in a tightly capped brown glass bottle. This solution was used to prepare sulphide series standard solutions for calibration of Sulphide and Cyanide electrodes and preparation also of sulphide solutions from spring waters. Also sodium sulphide was directly dissolved in boiled and cooled distilled water for determination of added and recoreved sulphide using iodimetric technique with sulphide and cyanide ion –selective electrodes.

Standardisation of prepared stock sodium sulphide solution in distilled water ⁽¹⁶⁾ :

The concentration of Na₂S stock solution was adjusted by potentiometric titration before proceeding with calibration of the ion –selective electrodes. The process involved titration of 25 mL of prepared Na₂S solution in the presence of sulphide or cyanide selective electrode with standard silver nitrate solution. The equivalence point was located for determining of sulphide ion concentration. Double junction calomel reference electrode .containing saturated potassium nitrate solution in its external compartment, was coupled with applied ISEs in each potentiometric determination.

Exploitation of iron filings :

The source of iron used for precipitation of sulphide from springs waters was obtained from the

residuals of sawing, drilling and cutting of steel rods, alloys and plates or ingot iron. These residuals were collected, washed from dust and impurities and dried. Accurate weight was taken and dissolved in hydrochloric acid (1:1) with warming on water bath until complete dissolving. The solution was cooled, filtered and the ppt was washed with distilled water and heated in an oven at 110 C⁰. The filtrate, most probable, contains iron(II) chloride was completed to certain volume with distilled water. 25 ml were transferred into conical flask and titrated with 0.1N KMnO₄ standard solution in the presence of 5ml of %85 H₃PO₄ until the solution becomes pinkish. The titration was repeated several times and the average was used to calculate the concentration of iron and its percentage in the iron filings which equals(93.40%). The ppt which is supposed to be whole carbon was dried, cooled and weighed to find its percentage in the original residual (6.2%).

Sampling of spring water :

Sampling should be of minimum aeration. Samples either analysed beside the springs or preserved for later analysis in the analytical laboratory. 4 drops of zinc acetate solution was added as additive to each 100 mL of sample. The sample bottles were completely filled and tightly stoppered to prevent the escape and oxidation of H₂S. Several volumetric flasks were half filled with 2M NaOH and 2% of NaNO₂ and completed to the mark with spring water samples. The solutions were stirred contiously and left for sulphide analysis by direct calibration method.

Standardisation of sodium thiosulphate solution⁽¹⁶⁾:

This solution was standardisd by titration with 0.1N KIO₃ solution in the presence of 10% of KI solution in acidic medium of 0.1M sulphuric acid until the solution became pale yellow. 1mL of starch solution was added and the titration was resumed to colourless. The titration was repeated four times and the concentration of Na₂S₂O₃ solution was adjusted.

Preparation and standardisation of 0.1N I₂ solution⁽¹⁶⁾:

12.6 g of I₂ was dissolved in 1L of ethanol (it solubility 17.3g/L) is in ethanol while 0.335 g / L in water) and standardised with 0.1N Na₂S₂O₃ standardisd solution using starch solution (1%) as indicator. ⁽¹⁷⁾

pH/mV – meter

Ion – analyzer type digital from Mettler – TOLEDO (China) was used to follow potential changes through potentiometric titration. The same instrument was used for pH measurements during titration and calibration modes.

Electrodes⁽¹⁷⁾ :

Two Ion – selective electrodes were used to monitor potential changes during titrations and calibration relative to Ag / AgCl saturated reference electrode with a precision of ± 0.1 mV as mentioned on the electrodes . These two electrodes were cyanide and sulphide solid – state membranes from Mettler – TOLEDO company.

A combined glass electrode from Mettler TOLEDO company was used to measure solution pH through calibration and titration with a precision of ± 0.1 pH unit as clarified by the company . The Ag / AgCl reference electrode of constant potential from Mettler TOLEDO containing 1M KNO₃ in its external compartment to avoid chloride ion interferences.

Magnetic Stirrer:

It is from minJank and kunkle company for stirring the solutions through calibration and titration processes.

Titration Cell:

It is a glass cell with cover of three openings two to introduce indicator and Ag / AgCl double junction reference electrodes and the third for introduction the end of the burette.

An atomic absorption flame spectrophotometer from phoenix 986 (USA): was used for determination of heavy metals in spring water . **A Flame photometer from Stuart Scientific (England):** was used for determination of alkali metals in spring water .

A HANNA Hi 2300 Micro processor conductivity meter from HANNA (Italian): was used for electrical conductivity measurements .

A HACH 2100 N Turbidimeter from HACH (Germany): was used for turbidity measurements .

Results and Discussion:

Calibration of sulphide and cyanide selective electrodes:

Sulphide and Cyanide ion- selective electrodes were calibrated in a series of iodide standard solutions ranged from 10^{-5} to 10^{-1} once in aqueous solution and second with 0.1 M KNO₃ solutions⁽¹⁹⁾ .The slopes and correlation coefficients were compiled in table (1) . Sulphide ion- selective electrode was calibrated also in a series of sulphide standard solutions prepared in SAOB . The results showed near Nernstian slopes and high correlation coefficients which means high response linearity of both electrodes. The efficiency of both electrodes were detected by following the titration of standard sulphide solutions in different concentrations with AgNO₃, FeCl₂ and I₂ standardised solutions. Table (2) illustrates the results of recovery of sulphide ion concentrations and the accuracy of the

results. Sulphide ISE showed high recovery from 96-98% with a negative percentage of errors which may be referred to loss of sulphide ions as volatile H₂S or adsorption on cell walls specially at low concentrations in addition to experimental errors⁽¹⁸⁾ .

Cyanide electrode gave lower recovery and high percentages of errors in addition to incapability of the electrode to follow the titrations with FeCl₂ and AgNO₃ solutions. This may be attributed to the sluggish of the electrode membrane as a result of its distortion and the appearance of pittings and micro bores on the membrane because of its frequent former uses and soluble complex formation with iodide ions^(19,20) . Therefore, most of the work was complemented using sulphide ion – selective electrode which gave very clear equivalence points and high jump in potential measurements in the range of 125-750mV/0.1ml.

Titration of spring water with AgNO₃, I₂ and FeCl₂ standardised solutions compared with calibration curve method⁽²¹⁾ .

The titration methods offered very close results for sulphide determination in spring water of AL-Kudhar spring for three seasons .But there were high variation in sulphide concentrations between the seasons table(3) . The exploitation of Asphalt of spring water has confused our work especially on spring and summer seasons where the Asphalt was manually removed and water was pumped by motor to its channels compared with natural elution of water in the past .

Therefore, high difference of sulphide concentrations especially on spring season where the sampling was carried out directly after Asphalt removing which gave opportunity for H₂S volatile in addition to elevated temperature on summer season which assists volatilization of hydrogen sulphide gas.

Local precipitating arrangement :

The first plan was to establish a concrete basin beside the spring water and spraying or dropping FeCl₂ solution from a net pipes above the basin .But the exploitation of the springs Asphalt have changed the plan in setting up local precipitating arrangement figure (1) .The arrangement consists of a tank (size 120 L) to contain a raw spring water withdrawn by water pump. Another tank was small (size 50L) containing 0.1N FeCl₂ solution prepared from iron filings and HCl. The third tank (size 20L) was the precipitating process which contains also mixer (water pump) to mix continuously spring water with FeCl₂ solution. The last tank (size 120 L) was the collective one which received the water and FeS from tank3 . The FeS is collected, filtered, washed dried, and casted into sticks to apply it in H₂S preparation and other sulphur

compounds . FeCl_2 solution prepared from iron filings and HCl (1:1) have shown high performance of extraction of sulphide from spring water reached 99.09% determined by sulphide ion – selective electrode

Physical and chemical properties of spring water:

Whole study of physical and chemical properties of spring water contents were carried out three times in winter, spring and summer. The study showed high concentrations of Cl^- , S^{2-} , Ca^{2+} , Mg^{2+} , Na^+ , K^+ total dissolved salts and high levels of hardness and alkalinity (Table4) . The concentrations of these contents were high and higher even after treatment, (Table5) . These higher contents of salts and hardness were arised from source of these spring water when they passed through limestone and dolomite in addition to stones changeable and solubility occurred under ground . Therefore these springs waters ar unsuitable for drinking, washing and agricultural purposes.

Heavy metals:

Determination of heavy metals : Cr, Ni, Pb, Co, Cd, Cu, Mn and Zn demonstrated high levels of these metals exceeded the permitted standard levels except (Zinc and Copper) which also increased the danger of using these springs waters (Table 6) . They are surprising results not found formerly . The high levels of these heavy metals may be arised from the new system of treating these springs . The process involved removing of Asphalt and water every 3-4 days to accelerate the elution of water and Asphalt Therefore, there is no sufficient time for these heavy metals to precipitate with sulphide ions as achieved formerly with natural elution of Ashphalt and water.

Conclusions:

Sulphide ion–selective electrode was successfully used to determine sulphide concentrations in Heat springs waters. The local precipitating arrangement offered high efficiency of extraction sulphide from spring water reached 99.09% using iron filing with HCl(1:1). The extracted FeS can be used as a source of H_2S in laboratories and other sulphur compounds in addition to reproduce FeCl_2 . which can be reused for precipitation process. High concentrations of hardness and total dissolved salts in addition to high levels of heavy metals in these springs waters devote more than one reason to render these spring water unsuitable for drinking, washing and agricultural purposes.

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Table (1) : slopes (mV /decade) and correlation coefficients of sulphide and cyanide ion-selective electrodes responses at25 °C.

The electrode	Sulphide – ISE	Cyanide - ISE
Slope aqueous	53.78±0.82	54.92±0.58
Correlation coefficient	0.9983	0.9982
Slope 0.1M KNO ₃	56.38±0.96	57.84±0.56
Correlation coefficient	0.9995	0.9999
Slope (SAOB)	27.46±0.67	-
Correlation coefficient	0.9988	-

Table (2) Results pf titration standard sulphide solutions in different concentrations with standardized AgNO₃, FeCl₂ and I₂ standardised solutions at 25 °C .

Compounds	The electrode	Temp °C	Added sulphide (ppm)	Recovery (ppm)	(%) Error	Potencial change at (ΔE)* eq point	pH at beginning titration	pH after the end of titration
Na ₂ S +AgNO ₃	Sulphide ISE	25	1516.8	1462.8	-3.56	750	13.2	10.1
		25	160	156.8	-2	503	11.64	9.4
		25	16	15.68	-2	348	9.28	6.5

Na ₂ S+ FeCl ₂	Sulphide ISE	25	25	1516.8	1476.8	2.64	-183	13.2	7.5
		25	25	160	155.2	-3	-125	11.64	7.04
		25	25	16	15.52	-3	-103	9.28	6.64
Na ₂ S+I ₂	Sulphide ISE	25	25	1516.8	1462.4	-3.58	-202	13.2	7.8
		25	25	160	155.2	-3	-166	11.64	7.1
		25	25	16	15.52	-3	-150	9.28	7.03
Na ₂ S+I ₂	Cyanide ISE	25	25	1516.8	1448	-4.5	-30	13.2	7.9
		25	25	160	153.6	-4	-22	11.64	7.13
		25	25	16	15.36	-4	-18	9.28	7.1

Table (3) : Results of sulphide ion concentration in AL-Kudhar spring in Heat city by titration methods compared with calibration curve method .

Calibration curve method in the presence of (SAOB)(ppm).	Titration with standardised FeCl ₂ (ppm).	Titration with standardised I ₂ (ppm).	Titration with standardised AgNO ₃ (ppm).	The electrode.	Month and year.
422.4±0.78	424.6±0.21	419.2±0.42	420.8±0.27	Sulphide ISE	2012 Decen ber
210.38 ±0.64	211.68 ±0.22	208.89 ±0.22	206.4±0.31	Sulphide ISE	2013 March
254.4±0.85	258.4±0.32	252.6±0.49	257.4±0.67	Sulphide ISE	2013 June

Table (4) : Physical and chemical properties of AL-Kudhar spring at three seasons at 25 °C

Parameters in mg/L unless otherwise stated	December winter 2012	March spring 2013	June summer 2013	Accepted value

T.S.S	T.D.S	Sulphide as S ²⁻	Potassium (as K ⁺)	Sodium (as Na ⁺)	Sulphates (as SO ₄ ²⁻)	Aluminum (as Al ³⁺)	Chloride (as Cl ⁻)	Magnesium (as Mg ²⁺)	Calcium (as Ca ²⁺)	Hardness (as CaCO ₃)	Alkalinity (as CaCO ₃)	E.C. μ S/cm 25 C	pH	Temperature e C ^o	Turbidity, NTU
105	20892	422.4	225	6200	35	BDL	10045	371	1108	4292	730	23666	6.55	18	30
119	22308	210	250	6310	46	BDL	13350	410	1131	4508	774	28726	6.42	27	39
125	21770	254	245	6256	38	BDL	12156	369	1116	4304	752	27945	6.33	36	36
	1000	0.2		200	400	0.2	250	50	150	500	125-200	2000	6.5 – 8.5	ACC.	5

Table (5) : Detection of spring water before and after treatment at 25 °C .

Potassium (as K ⁺)	Sodium (as Na ⁺)	Sulphates (as SO ₄ ²⁻)	Aluminum (as Al ³⁺)	Chloride (as Cl ⁻)	Magnesium (as Mg ²⁺)	Calcium (as Ca ²⁺)	Hardness (as CaCO ₃)	Alkalinity (as CaCO ₃)	E.C. μ S/cm 25 C	PH	Temperature e C	Turbidity, NTU	Parameters in mg/L Unless other wise stated
225	6200	35	NIL	10022	371	1108	4292	730	23666	6.55	17	30	Spring raw water
222.5	6125	43	NIL	10858	379	976	3993	749	26417	5.84	18	118	Treatment with standard FeCl ₂ solution
220	6150	52	NIL	11570	381	992	4042	753	25560	5.31	16	135	Treatment with iron filings
-	200	400	0.2	250	50	150	500	125-200	2000	6.5 – 8.5	ACC.	5	MPL

Sulphide (as S ²⁻)	422.4	4.96	3.84	0.2
T.D.S	21892	22363	22178	1000
T.S.S	105	190	208	.

(6) : Some heavy metals concentrations (ppm) in AL-Kudhar spring and at different seasons

Cr	Ni	Zn	Pb	Co	Cd	Cu	Mn	Fe	Month and year
0.90	0.52	0.07	0.79	0.81	0.14	0.09	0.07	0.59	December 2012
1.1	6.52	0.09	0.34	0.67	0.09	0.13	0.7	0.87	March 2013
0.7	6.73	0.11	0.36	0.83	0.07	0.1	0.63	0.93	June 2013
0.05	0.2	1	0.01		0.003	1	0.1	0.5	Standard boundaries

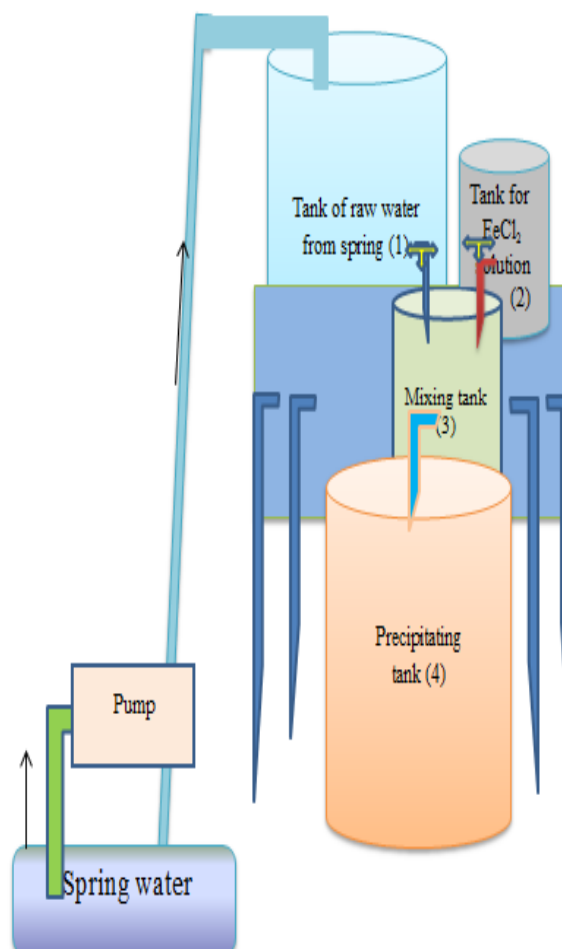


Figure (1) :The local arrangement of sulphide treatment.

استخلاص الكبريت من مياه العيون الكبريتية وإمكانية استثمار تلك المياه في الأغراض البشرية والزراعية

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الخلاصة

تضمن البحث تحضير منظومة لإستخلاص الكبريت من مياه عيون هيت الكبريتية لخطورته على صحة الانسان وسميته وتلويته للبيئة حيث استعمل كلوريد الحديد (II) المحضر من برادة الحديد وحامض الهيدروكلوريك (1:1) لإستخلاص H₂S على شكل FeS الذي يمكن الاستفادة منه في تحضير H₂S في المختبرات اضافة الى مركبات الكبريت الاخرى. اعطت التقنية كفاءة عالية في استخلاص الكبريت وصلت الى 99.09% حيث استعمل قطب الكبريتيد الانتقائي بنجاح في متابعة وتقدير تركيز الكبريتيد قبل وبعد المعالجة . اعتمدت عملية الاستخلاص بكلوريد الحديد(II) بسبب توفره ورخص ثمنه وسهولة استرجاعه. اثبتت دراسة الخصائص الفيزيائية والكيميائية لمكونات هذه المياه احتوائها على تراكيز عالية من العسرة والاملاح الذائبة حتى بعد المعالجة بمعدل 21656 جزءاً من المليون، لذا فإن هذه المياه غير صالحة للشرب والغسيل والاغراض الزراعية. كما ان وجود تراكيز عالية من العناصر الثقيلة تعدت المستويات المسموح بها قد زاد من خطورة استعمال هذه المياه.