A New Novel Derived from Sodium Carbonate for The Elimination of Hydrogen Sulfide from Biogas

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Abstract: Worldwide, the problems related to conventional energy resources can be overcome using renewable energy resources such as biogas. The availability of pollutants such as hydrogen sulfide that can harm the end user's health necessitated the researchers to find a way to eliminate the pollutant from the biofuel. The study uses sodium carbonate (soda ash) from Lake Natron to remove hydrogen sulfide from biogas. Different parameters such as particle size (280 and 400μm), the mass of sorbent (25, 50, and 75g), and biogas flow rate (0.04 m^3/h and 0.03) were considered in this study. Mineral composition, textural characteristics, and surface morphology of the sorbent were determined via X-ray, Quantachrome NOVA 4200, and Field Emission Scanning Electron Microscopy (FE-SEM) respectively. The high adsorption ability of 0.02g/100g and removal efficiency of 94% was noted in the reactor containing particle size 280μ, mass 75g, and biogas flow rate of 0.03m^3/h. The used-up sorbent was regenerated with great success for 1, 5, and 7 days considering that 7 days regenerated sorbent shows good performance with an adsorption ability of 0.01g/100g and a removal efficiency of 90% observed at the breakthrough point with 5 round/cycles. Its performance such as adsorption ability and high removal efficiency makes the material to be promising sorbent material for biogas purification.

Keywords: Biogas, Hydrogen Sulfide, Soda Ash, Regeneration

I. INTRODUCTION

Worldwide immediate measures are needed to resolve the problems related to the use of non-renewable energy resources that resulted in climatic change including acidic rain [1], greenhouse gas emissions [2], air pollution, and water pollution. Among the viable solution for the greenhouse gas emissions resulted because of the use of conventional energy sources, as oil is biogas, which is formed due to the AD of organic matter. Biogas is an eco-friendly and renewable energy that can have different applications such as replacing heat, vehicle fossil fuels, and power [3]. The quality and

quantity of biogas formed during the anaerobic digestion of the feedstock are determined by the quality and quantity of the feedstock used in the biogas digester. Municipal solid waste (MSW) and some residuals from kitchen waste are highly preferred substrates for biogas generation as they recover energy and resolve the challenges associated with waste management and disposal. Biogas composition relay on the feedstock substrate utilized in the bio-digester although mainly consists of CH_4 60-70%, CO_2 30-40%, hydrogen sulfide, ammonia, and nitrogen [4]. The purity and the concentration of methane are the principal criteria to be used in valuing the calorific value and the energy density of biogas which varies from 15-30 MJ.Nm^{-3} [5].

The existence of H_2S in biogas has a significant issue, mainly in the engine of the machine [6], metal parts, and animal health [7]. H_2S in a concentration ranging from 1000 to 3000ppm can lead to harm to humans in a way H_2S reacts with hemoglobin in the blood hence inhibiting respiration and thus leading to collapse, pulmonary paralysis, and lastly death [8]. The existence of sulfur compounds in combustible fuels like biogas is subject to increasing severe obstruction on the environment as it releases sulfur dioxide to the environment and thud climatic change. Various techniques for the elimination of hydrogen sulfide from biogas as reported in the literature. Fixed bed columns, slurry reactors, and liquid adsorption have been used in the desulfurization of natural gas with positive results [9]. Large scale industries especially for the removal of sour gasses the method adopted mostly is alkylamine, due to the reason that they are demanding high costs including energy use [10] [11]. The amount of H_2S from landfill (LAD) and that of anaerobic digestion from the bio-digester has been compared and it was noted that the quantity of H_2S in digester anaerobic digestion is almost 40 times greater than that in landfill anaerobic digestion. The elimination of hydrogen sulfide (H_2S) from biogas is required and it depends on the end use of biogas, for example, some application of biogas needs a high energy content biogas like vehicle fuel [12] and injection into the national grid where the concentration of hydrogen sulfide should be 0.5 to 10ppm[13]. The availability of hydrogen sulfide in biogas usually activate the deterioration of electrode and thus lowers the performance [14]. Two principals are used to operate in a fixed bed reactor for the desulfurization process such as physisorption and chemisorption. The establishment of a chemical bond between the adsorbate and the sorbent material

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is a chemisorption process [15] whereas the transfer of pollutant molecules to the sorbent material using a surface-based exothermic process is a physisorption process [16]. The weak Van der Waals forces and the electrostatic interaction are used to dominate the physisorption while chemical bond for example covalent bond between sorbent material and adsorbate is used to dominate chemisorption.

Lake Natron is situated at 36.0458° E; 2.3436°S in the Northern part of Tanzania, that is a small but localized Lake that has Soda ash (sodium carbonate salt) that can be utilized as a promising sorbent for the removal of hydrogen sulfide (H₂S) from biogas. When considering material for the desulfurization process then it is necessary to examine the pH of the sorbent before the adsorption process. The sorbent with an acidic pH is mostly dominated by (H⁺) on the sorbent surface that unfavor hydrogen sulfide to be adsorbed because of either competition of the adsorption surface of the sorbent material or repelling force. Therefore; when hydrogen sulfide dissociates will be hindered by the acidic pH of the sorbent material and therefore will limit the elemental sulfur to be oxidized [17]. The soda ash from Lake Natron has an exceptional property, as its pH is 12, and have a huge amount of sodium carbonate that can be used for the hydrogen desulfurization process. The current study demonstrates the effectiveness of soda ash sorbent that is a locally available natural and low-cost sorbent showing high performance in the sorption process and thus can replace the commercial sorbent for the desulfurization process. Reuse and regeneration of the soda ash material were done successfully five times which indicated the sorbent was appropriate and cost-effective for the desulfurization process.

II. MATERIALS AND METHODS USED

A. Equipment and materials

The used material in this study was a fabricated bed reactor, soda ash, cotton wool, a clear tube (1/16), sand as an inert material, and raw biogas. The equipment used in the current study were sieving machines, masking tape 50m×12 mm, aluminum foil, oven LDO-150), biogas analyzer Geotech 5000, grinder HK 820, electronic balance HZT-A200, and flow meter JBD2-5-SA.

B. Methods

Sorbent Gathering and Preparation

The sorbent was obtained from the Lake Natron 36.0458° E; 2.3436°S, ground, and sieved into two different particle sizes 280 and 400µm, packed with the addition of constant mass of inert material for the smooth passage of the gas, see **Fig 1(a-d)**. The mass of the inert material was 30g while the sorbent mass was 25, 50, and 75g.

(a)   (b)   (c)   (d) Fixed-bed reactor

Fig 1: Preparation of sodium carbonate sorbent for sorption purposes: (a) bigger particle size (b) grinding machine (c) working size (d) Fixed bed column.

Biogas sources

The adsorption capacity of the sorbent material was determined via an on-site experiment, which was conducted at the household premises, and the biogas analyzer was used. The biogas was from two digesters and their constituents vary because of the feedstock **Table 1**. Biogas were from kitchen waste remains, biomass, and human waste.

Sorbent performance

The uptake ability (SC) and the removal efficiency (RE) were obtained by the use of **equations 1** and **2 [18]**

$$RE = (\frac{C_O - C}{C_O}) * 100\% \tag{1}$$

$$SC = WHSV * [\frac{M}{Vmol} * \int_0^t (C_O - C)dt] \tag{2}$$

where V_{mol} is the gas molar volume (22.4 dm³) at standard conditions in L/mol, M is the atomic mass of sulfur, t is the time for operating, Co is concentration before the sorption process, C is concentrations of H₂S after the adsorption process in ppm, and WHSV is the weight hourly space velocity in mLh⁻¹g⁻¹, [19]. The regeneration reuse process and the chemical reaction are indicated in equations 3&4 [20].

$$Na_2CO_3 + H_2S \rightleftharpoons NaHS + NaHCO_3 \tag{3}$$

$$NaHS + 0.5O_2 \rightarrow S° + NaOH + Na_2CO_3 \tag{4}$$
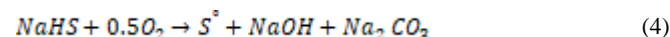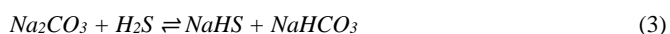
Characterization of the material

The three samples that are raw samples (S₁), spent (S₂), and regenerated samples (S₃) were examined for textual properties. The specific surface area was evaluated via Brunauer-Emmett-Teller technique whereas the distribution of the pore size was examined by Barrett-Joyner-Halenda (BJH) technique. The Quantachrome NOVA 4200 was utilized for the evaluation of nitrogen adsorption-desorption isotherms at 77K at the University of Dar es Salaam, Tanzania and the sample material was degassed before adsorption-desorption process for about 3hrs at 120°C. The raw sample (S₁), spent sample (S₂), and regenerated sorbent (S₃) were analyzed for pore volume, BET surface area, and pore diameter while the composition of sorbent was assessed by (XRF) energy-dispersive X-ray fluorescence spectrometer. The surface morphology was performed at the Indian Institute of Science

and Technology Bengaluru, India via Field Emission Scanning Electron Microscopy (FE-SEM).

Performance and Adsorption Evaluation

Cotton wool was filled up in a plastic bed reactor with a 4 cm in diameter and height of 13 cm, in the absence of sorbent material biogas was allowed to flow in at room temperature to be confident that cotton wool can react with biogas or not. It was noted that hydrogen sulfide does not react with cotton wool, as the outlet concentration was the same as the inlet concentration. The soda ash sorbent was then mixed with sand as inert material for the smooth passage of the biogas in the reactor. The soda ash sorbent was packed in a bed reactor, both ends were held with cotton wool, and biogas was authorized to pass. Before the adsorption process, the amount of hydrogen sulfide in biogas was recorded once and then monitored after 15 minutes. An illustration and photo from the on-site adsorption process are indicated in **Fig 2(a, b).**
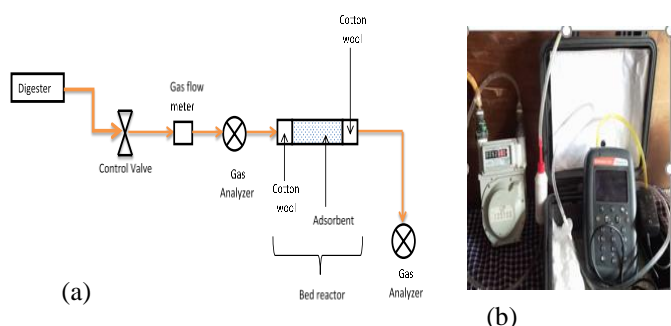

(a)

(b)

Fig 2: Purification experiment: (a) An illustration (b) Photo

Sorbent regeneration

Sorbent regeneration was done to investigate the uptake ability and removal efficiency of the used soda ash in comparison to the original sorbent. The spent sorbent was taken off from the fixed reactor and laid out in a clean sheet to display it to the atmosphere and was performed following equation. It was conducted for 1, 5, and 7 days of exposure which is in line with [21], the sorbent sample was then returned to the fixed bed reactor and the adsorption process goes on.

III. RESULTS AND DISCUSSION

A. The Composition of On-Site Biogas

Biogas analyzer model Geotech 5000 was used to analyze the raw biogas from the two digesters and the outcomes are as tabulated in **Table 1**. The constituents of the biogas from the two bio-digesters differ because the feedstock used in the bio-digester was different day after day.

TABLE I: Composition of on-site biogas

Composition	Digester I	Digester II
Methane (v.%)	67-69.1	69-70.1
Carbon dioxide (v.%)	29.7-30.1	30.1-30.8
Oxygen (v.%)	0.1-0.2	0.1-0.2
Ammonia (ppm)	331-421	331-530
Hydrogen sulfide (ppm)	530-606	748-1480

B. Textural properties of the sorbent

The three samples were examined for textural characteristics as a raw sample, spent, and regenerated sample. Isotherms corresponding to adsorption-desorption were drawn from the data as illustrated in **Fig 3a** whereas pore size distribution obtained via BJB techniques is illustrated in **Fig 3b.** Taking into consideration **Fig 3a**, more nitrogen has been adsorbed by unspent sorbent material (S_1), and a high surface area has been noted in **Table 2**. Sample S_1 shows a high differential volume due to a large pore radius, which contributed due to the large surface area in comparison to other samples **Fig 3b**. Literature show that the greater the area the more adsorption site available for hydrogen sulfide to be attached [22]. **Table 2** indicated the multipoint BET evaluation for the three sorbent materials and was noted that the surface area for the unspent sorbent sample was higher than spent and regenerated sorbent. The BET surface area was lastly reduced for the sample that undergo adsorption (S_1) due to the reason that the mesoporous adsorption site has been occupied by the pollutant (H_2S) from biogas. The pore size obtained from both samples indicated that they are more mesoporous rather than microspores and macroporous as the size for three samples all relay from 2-50nm, which is in line with literature data [23] [24].
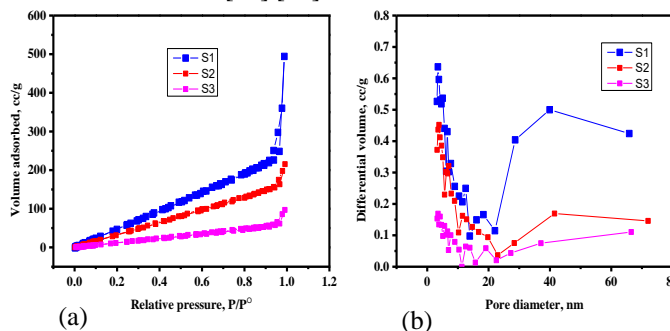

(a)

(b)

Fig 3: (a) Isotherms from nitrogen adsorption/desorption (b) Pore size distribution obtained via the BJH technique.

TABLE II: Multipoint BET data for S_1 (unspent sample), S_2 (spent sample), and S_3 (regenerated sample) particle size 280μm.

Samples	$S_{(BET)}$ (m^2/g)	Pore radius ($A°$)	V(total) (cm^3/g)
S_1	409.4	17.7	0.7
S_2	264.2	17.6	0.3
S_3	103.1	17.4	0.1

C. Morphology of the sorbent

Evaluation of the sorbent via SEM was carried out on the three samples S_1, S_2, and S_3 to observe the different in structure for the three samples. The SEM images for unspent material spent material and regenerated were shown as illustrated in **Fig 4(a-c)**. The surface area for the unspent sample seems to be relatively rough with some hollows, the crystal structure was irregular thus high porosity enhances the adsorption of H_2S from biogas **Fig 4(a)**. The smoothness of the sorbent and some masses of sorbate were observed to be attached on the surface of the spent sorbent as observed in **Fig 4b** and thus the porosity of the spent sorbent was reduced indicating there is some foreign material attached that is hydrogen sulfide from

biogas. **Fig 4(c)** shows a regenerated and reuse sorbent with some masses on the hollows as observed on the micrograph showing there are attachments of hydrogen sulfide.
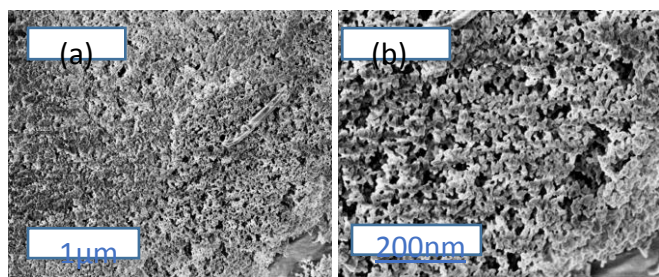

Fig 4 (a): Scanning Electron Microscope images for unspent sorbent S_1 (a) 10 KX (b) 25 KX
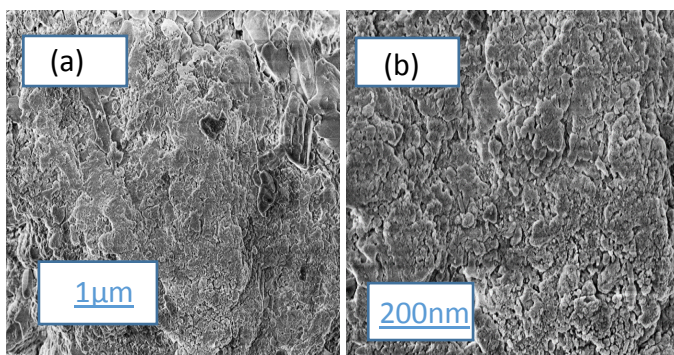

Fig 4(b): Scanning Electron Microscope images for unspent sorbent S_1 (a) 10 KX (b) 25 KX
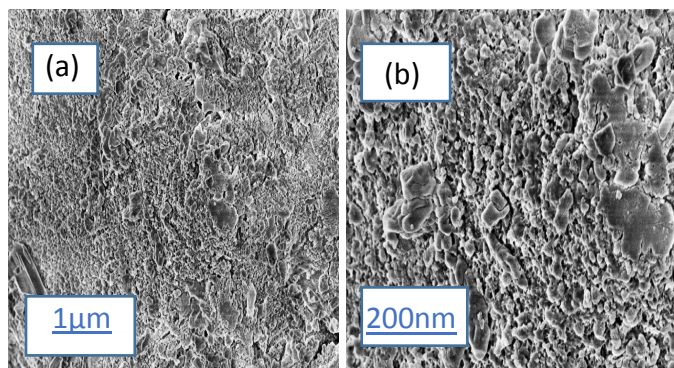

Fig 4(c): Scanning Electron Microscope images for unspent sorbent S_1 (a) 10 KX (b) 25 KX

D. pH and mineral constituents of soda ash adsorbent

Table 3 illustrate elements contained in soda ash sorbent via XRF analysis and was noted that Iron, sodium carbonate, Chlorine, Calcium, Zirconium, and Potassium were the most element present in the sample in huge amount. The soda ash pH was noted to be basicity (12) that favors the adsorption of acidic sorbate which agreed with the literature [25]. The availability of a huge amount of sodium carbonate compound in the sorbent material seems to enable the hydrogen sulfide uptake from biogas as observed in **equation 3.**

TABLE III: Elemental composition of the sorbent

Element/ compound	Si	NaCO$_3$	K	Cl	Fe	Ca	Zr
Amount (mg/L)	965. 68	21140 7.7	106 1.08	104 84.8	247. 6	735. 1	191 .86

E. Adsorption capability of soda ash sorbent

Various parameters such as gas flow rate (FR), particle size, and mass of adsorbent were used in the evaluation of the adsorption ability of soda ash in the elimination of H_2S from biogas. Hydrogen sulfide from biogas reacts with soda ash and form sodium bicarbonate and sodium hydrogen sulfide as per **equation 3**. The occupation of the mesoporous site of the sorbent by hydrogen sulfide from biogas makes the sorbent saturated at a specific time depending on the nature of the material [26]**.**

F. Effect of Sorbent Mass

In studying the effect of mass on the removal of H_2S from biogas, masses of sorbent were varied whereas the volume of biogas, hydrogen sulfide concentration, and particle size of sorbent were maintained constant. The uptake and removal of H_2S from biogas is as illustrated in Fig **5(a, b)**. An increase in sorbent mass resulted in the rise in sorption capacity and removal efficiency of the sorbent material while breakthrough was delayed, based on the fact that there is an increase in adsorption site for adsorption which is in line with [27] [28] [29]. It is because there is an enlarged interfacial external surface area for biogas to interact with the adsorbent material that leads to an increase in sorption capacity, removal ability, and saturation time. For a sorbent, to be durable before regeneration and reuse there is a need for having a long and large bed reactor that can allow for more mass of sorbent. Literature shows that sweet potato leaves with 1kg mass showed a sorption capacity was 0.37 g/100 g whereas removal efficiency was 95% [18], while Mrosso et al [30] use the mass of 0.6g for the elimination of H_2S from biogas that showed a sorption ability was 0.37 g/100 g and removal efficiency RE 95%. Based on the current study, it was noted that H_2S molecules had more chances to interact with 75 g of soda ash in comparison with 50, and 25 g. Thus the highest RE and Sc were noted when using a mass of 75g.
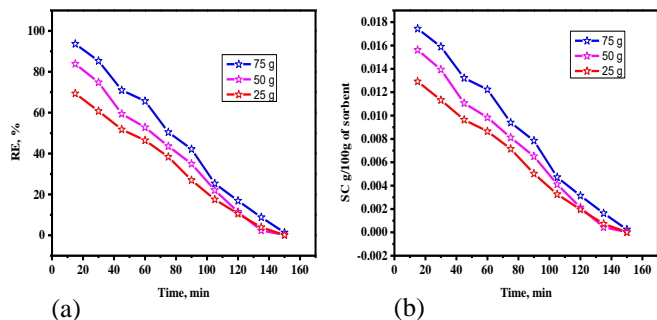
(a)　　　　　　　　　(b)

Fig 5: Performance of soda ash on the effect of adsorbent mass (a) RE (b) SC. Conditions for evaluation were Co=1227ppm, Flow rate 0.03m³/h and size was 280 µm.

G. Effect of Particle Size

The material particle size is an important aspect to be considered when conducting adsorption experiments, thus to improve the adsorption ability the particle size of the material is of significance in the kinetics of adsorption and thus must be put into consideration as the adsorption rate is inverse to particle size [31]. The % removal and uptake of H_2S are determined by the size of the sorbent, removal efficiency (RE) decreases with the increase in sorbent particle size, and mass transfer enhances the uptake capacity of sorbent [32]. Fig sorbent with small size showed higher adsorption ability than large size which might be contributed by high surface area to volume ratio **Fig 6(a, b)**. Literature explored that small particle size provides more exposure to the sorbent for the adsorption process, which resulted in high SC and removal efficiency. A study done on the purification of biogas indicated that the purity increased from 87.7-90% as the particle size decreased [33]. The same was found in the current study where 280 µm shows a SC of 0.02g/100g whereas RE of 94% within the first 15 minutes.
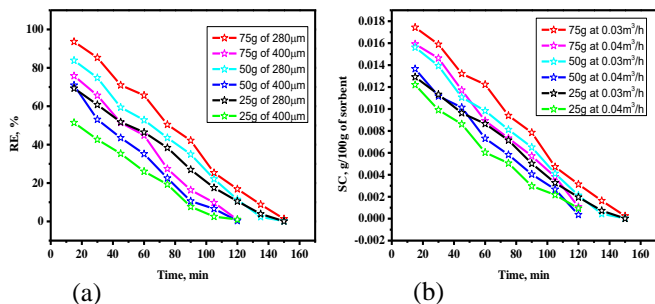

(a)　　　　　　　　　(b)

Fig 6: Performance of soda ash on the effect of particle size (a) RE (b) SC. Conditions for evaluation were Co=1228ppm, Flow rate 0.03m³/h and size was 280 µm.

H. Effect of flow rate on sorbent performance

The flow rate of biogas had a great influence on the adsorption and uptake of H_2S from biogas. As the flow rate decreased then the uptake of H_2S from biogas increases because there is more time for contact between adsorbate and adsorbent and vice versa [34]. When a high biogas flow rate is used, the time for interaction between the hydrogen sulfide and soda ash decreased and thus biogas passes the soda ash

without adsorbed which leads to low SC and removal efficiency. Two different flow rates were used to evaluate the outcomes of the biogas flow rate on the sorption ability of the sorbent as of 0.03m³/h and 0.04m³/h where a low flow rate showed a high RE of 94% and SC of 0.02 g/100g of sorbent which is in line with the literature [35]. Therefore, the uptake and elimination of H_2S from biogas rise as the interaction time between the adsorbate and sorbent is enough for the hydrogen sulfide to diffuse into the mesoporous sites of the sorbent Fig **7(a, b)**.
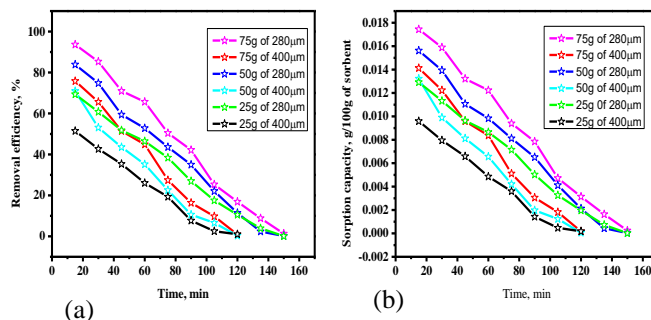

(a)　　　　　　　　　(b)

Fig 7: Performance of soda ash on the effect of flow rate (a) RE (b) SC. Conditions for evaluation were Co=1480ppm, Flow rate 0.03m³/h and size was 280 µm.

I. Sample regenerated and its performance

Before the selection of sorbent for purification purposes, it is crucial to evaluate its regenerative ability. The spent sorbent was regenerated and reused for 1, 5, and 7 days of exposure, and a comparison between the original sample and the regenerated as made. The results approved that the original sample performed well as compared to the regenerated and reused samples. The time for sorbate to contact with sorbent was higher in the original sample 150 minutes than in regenerated sorbent. The regenerated sorbent for 7 days exposure took 120 min to saturate for 1st -3rd cycles while in the 4th-5th round/cycle, the material saturated at 105 minutes. The time for interaction decreased in the last two cycles indicating that the material loses its ability to uptake pollutants from biogas. In the first 15 minutes for the first round/cycle, the material showed a RE and sorption capacity of 90%, and 0.016/100g respectively whereas at 60 minutes RE and Sc of 52% and 0.01 g/100g were noted. Based on the results, it was noted that as you move from lower cycles the uptake of hydrogen sulfide from biogas decreases **Fig 8(a, b)**.
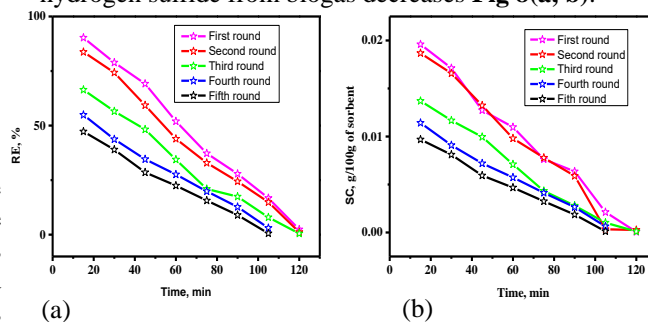

(a)　　　　　　　　　(b)

Fig 8: RE and SC of regenerated/reused soda ash sorbent: Particle size 280 µm, Flow rate 0.03m³/h, days of exposure days 7.

During five days of exposure, the first round took 120 minutes for the sorbent to saturate while 2nd -the 4th round saturate after 105 minutes Fig 9(a, b). The removal efficiency for the first 15 minutes of the first round was observed to be 78% whereas at 60 minutes was 48%. A sudden decrease in the achievement of soda ash on the removal and sorption ability was noted for 1, and 5 days of exposure which might be due to the inability to recover all adsorptive sites. The results from regenerated sorbent for both 1, 5, and 7 days of exposure during the first 15 minutes proved that the sorbent is a promising material as it produces a RE of greater than 50%.
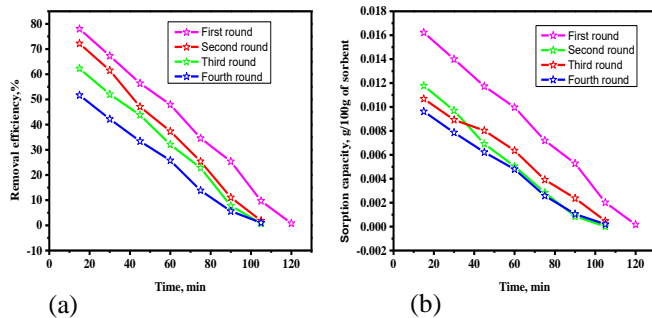

Fig 9: RE and SC of regenerated/reused soda ash sorbent: Particle size 280 μm, Flow rate 0.03m³/h, days of exposure days 5.

One-day exposure indicated less contact time between the sorbate and adsorbent material which resulted in low performance and contributed to the fact that all the sites could not be recovered [36]. The RE and SC of 1 day of exposure are as illustrated in **Fig 10(a, b)**. The sorbent loses its ability as it adsorbs for a while, was proven by the results obtained from the present study, and thus seven days of exposure resulted in higher removal efficiency and sorption in comparison to one and five days of exposure.
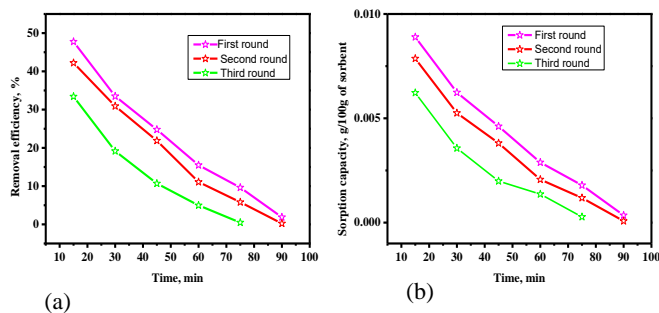

Fig 10: RE and SC of regenerated/reused soda ash sorbent: Particle size 280 μm, Flow rate 0.03m³/h, days of exposure days 1.

J. A contrast of soda ash with other materials

Table 5 indicates a comparison made between the present study and previous studies. The material used in this study illustrated a high performance with other materials and thus an appropriate sorbent for the desulfurization process.

TABLE V: A contrast between the present works with previous work

Adsorbent	Flowrate used (m³/h)	Time used (h)	Re (%)	References
Red Rock (Nadosoito)	0.006	2.5	95	[30]
Sweet potatoes leaves	0.020	1.2	95	[18]
Oldonyo Lengai ashes	0.120	1.5	96	[37]
Fe/EDTA	0.016	0.8	84.5	[38]
WHAC-1:1-650	0.024	2.0	93	[39]
Red mud soil	0.003	1.5	-	[40]
Soda ash	0.030	2.5	94	This work

IV. CONCLUSIONS

In this study, soda ash was used as an adsorbent for the desulfurization process at room temperature. XRF analysis indicated that among the mineral present sodium carbonate were present in huge number than other elements and thus the one that enables the adsorption process. The performance of soda ash was tested via an on-site adsorption experiment and it was proved that small particle size280 μm, a large mass of 75 g, and a small flow rate of 0.03m³ indicated high performance with a RE of 94% and SC of 0.02g/100g. Meanwhile, the study succeed in regenerating the sorbent for different days, as 1, 5, and 7 days whereas 7 days of regeneration exhibit a good performance with RE 90% and SC was 0.01g/100g.

V. DATA AVAILABILITY

Data used to support these findings are available when they are required.

VI. ACKNOWLEDGMENTS

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