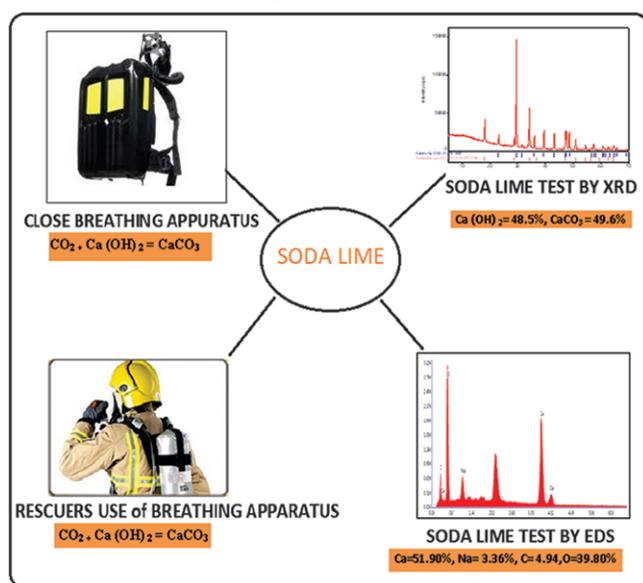


Critical analysis of Indian soda lime: scope of improvement

Graphical abstract



Soda lime is one of the most popular carbon dioxide absorbent materials to be used for closed-circuit life saving safety breathing apparatus in mining industries. A trained rescue person uses it during a situation such as a fire, explosion or emission of toxic gasses in underground mines. This paper evaluates the chemical composition and physical properties of soda lime using specific parameters (moisture, carbon dioxide gas absorption, granule shape and fine particle size) which plays an important role in its application in breathing apparatus. Results indicated that soda lime moisture content, fine grains and hardness ranged between 11.6-18.3%, 0.2-1.9g, and 70-90%, respectively. The CO₂ absorption rate was observed to be 20.0 to 57.0 minutes compared to standard UK Protosorb soda lime CO₂ (135 minutes). X-Ray Diffraction (XRD), Energy Dispersive Spectroscopy (EDS) and Scanning Electron Microscope

(SEM) analysis of the samples were carried out to understand the changes in molecular structure of the material before and after CO₂ absorption. The XRD result indicated presence of portlandite (48.5%), calcite (49.6%) and potassium rhenium sulfide telluride cyan acetate (PRSTCA) (1.84%) before CO₂ absorption and calcite/calcium carbonate (89.4%) portlandite (3.38%) and octasodium d-potassium tetra hydrogen dihydroxo tetra telluride dipalladate (7.2%), 20-hydrate was observed after CO₂ absorption. EDS of sample 6 indicated presence of carbon (4.94%), oxygen (39.80%) sodium (3.36%) and calcium (51.90%) before CO₂ absorption and carbon (6.27%), oxygen (36.96%), sodium (1.37%) and calcium (55.40%) after CO₂ absorption.

Keywords: Carbon dioxide (CO₂), soda lime, hot air oven, breathing apparatus, XRD, EDS, SEM.

1. Introduction

The revitalization of air present in the atmosphere of all nature is of great importance. This applies to any type of atmosphere, be that of a space capsule, a submarine or any enclosed compartment [1]. The atmosphere factor within the enclosed system is vital for life, and hence air purification has been introduced to remove contaminants or any types of toxic gases, in order to maintain a healthy air composition. For example, an excess amount of carbon dioxide should be removed, and this can be done either by a chemical separation technology or a physical absorption process [2, 3].

The air purification system employed should be capable of maintaining appropriate CO₂ and oxygen levels so that it will not damage mental and physical performances of a human being. At 0.04% CO₂ content, the enclosed environment must be ventilated, and preparations made to remove the gas [4, 5]. CO₂ absorbent materials are well-known and widely used in a number of applications. They may include, for example, many kinds of industries, such as anesthesia, military, and submarines, fire protection apparatus, and mine rescue apparatus. Soda lime is a mixture of calcium hydroxide, sodium hydroxide and water.

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Soda lime, the most popular CO₂ absorbent material, was first discovered by Robert Erastus Wilson of the Massachusetts Institute of Technology (MIT) in 1917 to meet the urgent need for an effective absorbent in military gas masks [6]. Under atmospheric CO₂ wet conditions, soda lime reacts and converts into CaCO₃. Soda lime is an absorbent material which contains a certain composition of calcium hydroxide (Slaked lime), metal oxides and moisture contents. As it reacts with CO₂ it forms a solid which is retained within the absorbent container [7]. It is white or a gray-white granule which rapidly deteriorates on coverage to air through absorption of carbon dioxide. It is kept and stored in a tightly closed container. It is used in a grainy form in a closed circuit breathing environment. Grain size, shape, moisture, hardness, activity and elemental composition are important in terms of absorbency. Variations in conditions, such as moisture, can significantly affect the predicted life expectancy and performance of the absorbent [8, 9]. A different key factor in determining absorbent performance is the grain size distribution. The grain size affects speed of reaction, which in turn affects the volume of the reaction zone and the capacity of the absorbent. However, smaller grain size, induces a greater pressure drops across absorbent, which can change absorption efficiency and other characteristic [10].

In an underground mining conditions there is a great concern towards safety and health conditions of workers and rescue operators are concerned. Miners should be able to work in the mines in a congenial environmental conditions without compromising with their health [11]. The environmental state should not harm their working condition. Self-contained closed circuit breathing apparatus are used in the adverse mining condition for rescuing the workers in fragile conditions. These apparatus need to supply oxygen for a specific time and allow CO₂ to be absorbed. Soda lime is helpful to achieve this. Self-rescuer apparatus are a type of breathing apparatus which not only provide oxygen, but also removes noxious gases such as carbon monoxide, carbon dioxide from the atmosphere to be breathed. It is a statutory requirement for a mine rescuer to carry out breathing apparatus with them for unfavourable conditions.

The aim of this study is an assessment of the soda lime quality. Soda lime is selected from different manufacturers to describe its physical properties, grain size, hardness, moisture and activity time. Data obtained will be compared to Indian standards of a similar result.

2.0 EXPERIMENTAL METHODOLOGY

Representative soda lime samples were collected from a three industrial manufacturers from three places of India (Ahmadabad, Kanpur, and Dhanbad) and a producer from UK. Five soda lime samples from each place are obtained to carry out the experiments. The five samples were aggregated to make one sample. Each sample was analysed in triplicate and mean values were considered. The Indian mining industry

follows IS: 5321, 1969 for estimation of different parameters that are described below:

2.1 MOISTURE

The loss on drying of moisture test was conducted using crucible and, hot air oven maintained at 150±2°C and desiccator containing freshly activated silica gel .5g Soda lime samples (in triplicate) were taken in the crucible. It was kept in a hot air oven for 2 to 2.5 hours at 150°C. The crucible was removed from the oven and cooled in a desiccator which had freshly activated silica gel for 30 minutes. Its weight was then measured using an electronic balance.

2.2 SIZE GRADING

The particle size measurement was undertaken by sieving using sieves of 200mm diameter and 2.80mm, 2.0mm, 1.40mm, +600um, -600um sieve sizes. 100g of the sample was taken in the sieve of 2.80 mm size and it is shaken for 5 minutes using a shaker machine. The shaker is operated at 285±3 cycle/min and then removes the sieves from the shaker machine and soda lime remaining on each sieve was collected and its weight was taken. The shape and length were measured using a dogmatic caliper

2.3 HARDNESS

The hardness of the sample was determined by shaking 50g of sample on a sieve of 2.00mm mesh size with 15 steel balls of 8 mm diameter. Cover the pan and shake in a shaker for 30 minutes, at the rate of 285±3 cycles/minute. After shaking the iron balls are removed carefully and brush any particles back into the pan. Transfer all the materials in the pan to a 1.40 mm sieve and shake for 3 minutes in the shaker. The material remained in the sieve is transferred to a petrie dish and took the weight as per BIS code IS: 5321, 1969.

2.4 CARBON DIOXIDE ABSORPTION PROPERTIES

This was evaluated by measuring the time taken by carbon dioxide to penetrate a soda lime column under prescribed conditions of the test as per (IS: 5321, 1969). Fill the absorption tube with soda lime as received. The tube shall be filled in by adding 95g of soda lime. It should be tightly packed, giving no open space to be filled with air. Air at the rate of 3 liters/min and carbon dioxide with a flow rate of 150cc/min controlled by flow meter is passed through a humidifier. Through water, saturator bubbler contains at 20°C to bring the relative humidity of the gas stream to at least 95 per cent before going through the absorption tube. The ambient air during the period of the test shall be at a temperature of 20°C±2°C and pass the air from the absorption tube through a bubbler of sodium hydroxide and barium chloride solution. When the soda lime fails to absorb the whole of CO₂ in a gas stream the solution in bubbler would become turbid, immediately barium chloride and sodium hydroxide solution is observed, connect a fresh bubbler and, if the solution becomes turbid in two minutes, the endpoint shall be considered to have been reached. The time from the commencement of the test in the beginning of the two minute

TABLE 1 VALUES OF GRADING SIZE DISTRIBUTION OF SODA LIME

Sieve size		2.8mm	2.0mm	+600 μ m	600 μ m
Sample Name		%	%	%	%
1	Supremesorb 1	2.5	70.0	1.0	1.0
2	Lisasorb	1.0	39.50	20.0	1.0
3	TRD International 1	1.0	50.1	14.3	0.8
4	Lifsorb	0.51	8.6	12.5	0.8
5	BEE Chems	13.0	19.5	6.0	0.2
6	TRD International 2	1.0	40.0	7.0	0.8
7	Supremesorb 2	1.9	70.5	2.5	1.9
8	UK Protosorb	2.1	30.0	9.0	0.7
	I.S. Permissible value	1.0	40.0	10.0	1.0

period shall be the absorption time. If the solution is not turbid at the end of the two minute period, connect another bubbler and repeat the process until occurs in two minutes. The total time taken in becoming a barite solution turbid is the activity reading.

2.5 CHARACTERIZATION OF SODALIME BY XRD (X-RAYDIFFRACTOMETER)

The soda lime material was characterized by using X-ray diffractometer (XRD) Model ultimo (iv) Rigako Japan. The measurement condition Goniometry is ultimo iv and attachment sample rotation at, scanning modal is 2 theta/theta and scanning type is continuing scanning. X-ray40kV/40mA, Cu wavelength is 1.5406A, DivSlit 2/3 deg, K-beta filter yes, start 5, stop 70, step 0.02, speed 10 deg/min.ultimo iv diffractometer to measure crystal planes that are perpendicular to the sample surface. This upgrade system can be especially useful to the characterization of the surface of a fill, the recording of a reciprocal lattice map.

2.6 CHARACTERIZATION OF SODA LIME USING BY ENERGY DISPERSIVE SPECTROSCOPY (EDS) AND SEM

EDS (energy dispersive spectroscopy) (Merlin/VP Compact Model) was used for the elemental study of the sodalime sample. It relies on dealings of a few sources of X-ray excitation and sample and scanning electron microscopes (SEM). EDS also helps to measure the multilayer shell thickness of metallic coating and analysis a combination of alloys. Many elements will overlap X-ray emission peak $Ti k_{\beta}$ and VK_{α} , $Mn k_{\beta}$ and $Fe k_{\alpha}$. Metal coating of sputtered gold/palladium for SEM imaging

3.0 Result and discussions

Results indicated that soda lime pH and temperature were 12.2 and 29.7°C. The physical appearance of soda lime granules is shown in Fig.1. The soda lime granule shapes were non-uniform, irregular and the length and the diameters varied between 2.60 to 6.11mm and 1.58 to 2.91mm, respectively. The current size distribution of the granules were considered as an optimum and got ability to resist air flow to enhance the soda lime absorptive capacity.



Fig.1 Granule size of soda lime

The diameter ranges (2.8mm, 2.0mm, 1.4mm, +600 μ , -600 μ) are shown in Fig.2. Results indicated that grain size of samples 1, 3, 7, 8 of 2.0mm satisfied and samples 1, 5, 7, 8 of 2.8mm did not satisfy the BIS Standard. The grain size of sample 2, 3, 4 of +600 μ m and sample 7 of 600 μ m, respectively were not fulfilling the BIS standard. The fine particle size of samples 1, 2, 3, 4, 5, 6, 8 of 600 μ m, samples 2, 3, 4, 6 of 2.8mm, samples 2, 4, 5, 6 of 2.0mm and sample 1, 5, 6, 7, 8 of +600 μ m satisfied the BIS Standard. The fine particle size (-600 μ m) showed relative distribution of weight of different samples 0.2%, 0.8%, 1%, 1.9%, 0.7% (Fig.2). The presence of average fine particle (0.92%) in the all samples were below 1%. Less

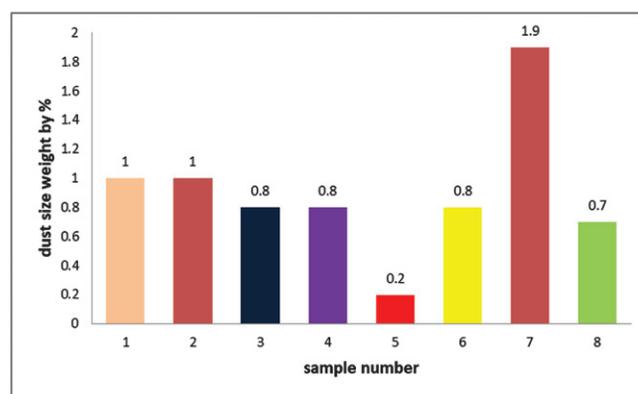


Fig.2 Average relationship between specified granular per cent by weight

fine particles and less sharp edge provided more comfortable and smooth running of the breathing apparatus.

The values of moisture content ranged from 11.6% to 18.3% and samples 1, 2, 4, 6, 7 and 8 were fulfilling the criteria of BIS values (16-19%) (IS: 5321, 1996). (IS Standard) except sample 5 (Fig.3) and values are given in the Table 2.

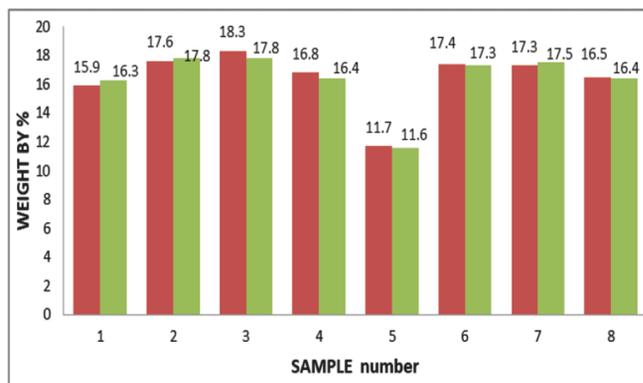


Fig.3 Variations of average moisture % by weight

TABLE 2: VARIATIONS OF SODA LIME MOISTURE IN DIFFERENT SAMPLES

Sample Name	Moisture		IS:5321 permissible value	
	Weight ₁ %	Weight ₂ %	Min.%	Max. %
1 Supremisorb 1	15.9	16.3		
2 Lisasorb	17.6	17.8		
3 TRD International 1	18.3	17.8		
4 Lifisorb	16.8	16.4		
5 BEE Chems	11.7	11.6	16%	19%
6 TRD International 2	17.4	17.3		
7 Supremisorb 2	17.3	17.5		
8 UK Protosorb	16.5	16.4		

The hardness test (BIS Standard) is presented in the Table 3. and compared to the permissible value of 70. The values of hardness varies from 70.0% to 96.1% and all samples were fulfilling the criteria of BIS (Fig.4).

Carbon dioxide absorption test (activity) is one of the most important parameters for soda lime quality evaluation. Results indicated that samples 1, 4, 6, 7 and 8 were fulfilling

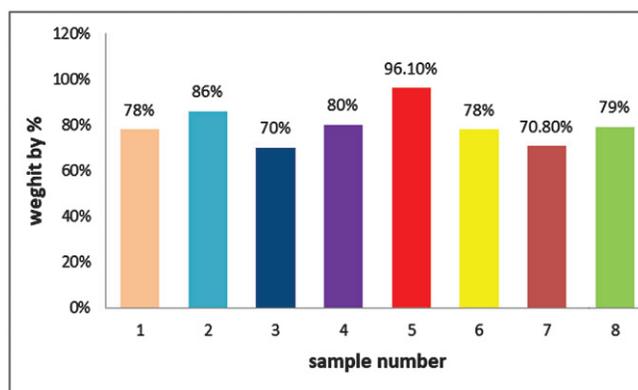


Fig.4 Variations of hardness (%) in different samples

the BIS standard and values were found less than 55 minutes except samples 2, 3 and 5. The results of the chemical absorbency varied from 20m to 135m (Fig.5), and achieved the CO₂ absorption specified by BIS (IS: 5321, 1996). The samples 1, 4, 6, 7, 8 exhibited better absorption capacity compared to samples 2, 3, 5.

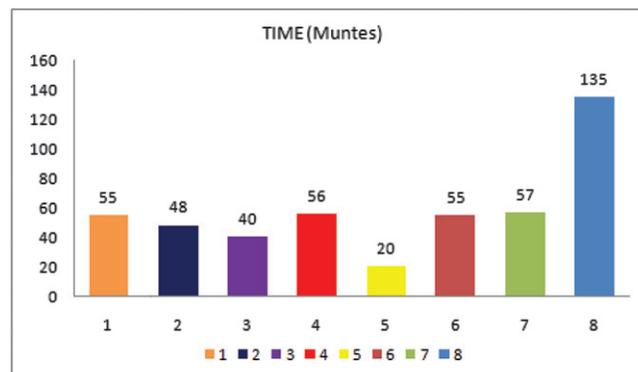


Fig.5 Variations of average activity, (minutes) of different samples

The British soda lime is considered to have the most absorption capacity fulfilling the criteria of activity more than 135 minutes (Fig.5).

3.1 ENERGY DISPERSIVE SPECTROSCOPY AND SEM ANALYSIS SAMPLE NO.5 BEFORE ABSORPTION

The main metal oxides determined by the EDS were calcium 63.16%, sodium 0.12%, silicon 2.25%, aluminum 4.15%

TABLE 3: VARIATIONS OF HARDNESS AND ACTIVITY IN DIFFERENT SAMPLES

Sample Name	Activity (minute)	Permissible value	Hardness (% by weight)	Permissible limit
1 Supremisorb 1	55	55m	78.0	70
2 Lisasorb	38		86.0	
3 TRD International 1	35		70.0	
4 Lifisorb	56		80.0	
5 BEE Chems	20		96.1	
6 TRD International 2	55		78.0	
7 Supremisorb 2	57		70.8	
8 UK Protosorb	135		79.0	

and oxygen 30.32% before CO₂ absorption and calcium 37.07%, sodium 1.22%, silicon 1.54%, aluminum 14.35%, gold 13.6% and oxygen 37.07% after CO₂ absorption.

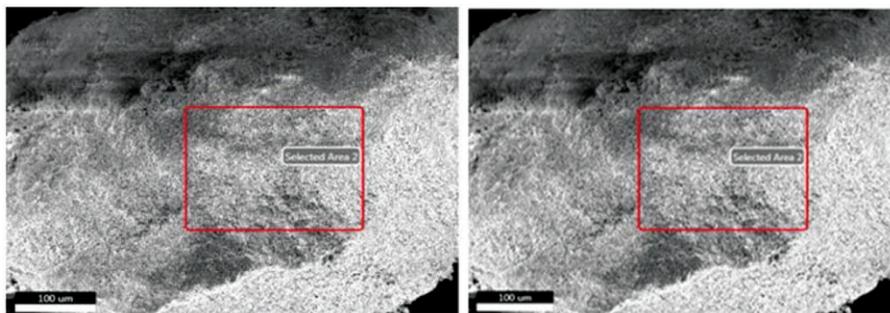
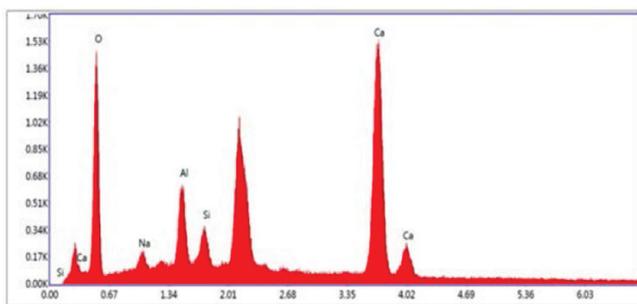
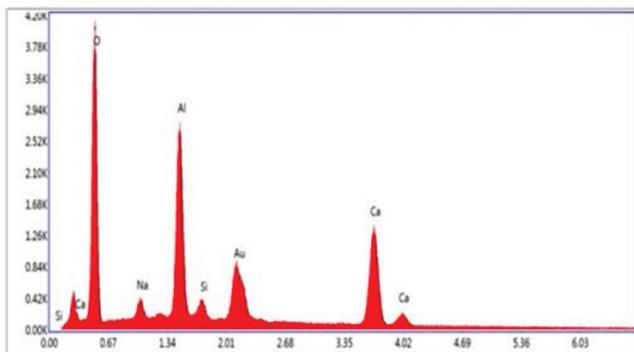


Fig.6 Scanning electron image of sample no.5 before and after CO₂ absorption



Lsec: 30.0 Cnts 0.000 keV Det: Octane Super Det



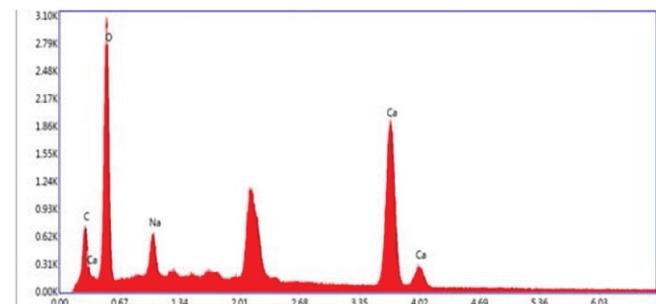
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Fig.7 EDS of sample no.5 elemental compositions before and after CO₂ absorption

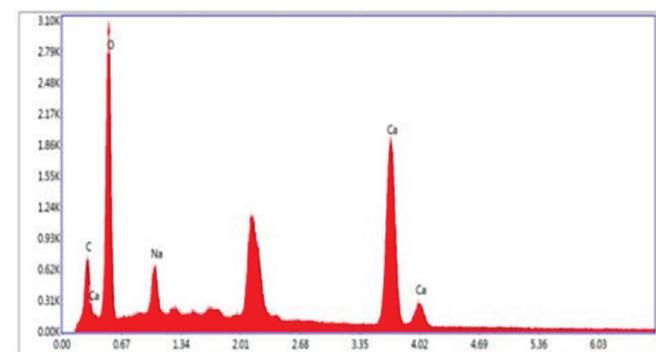
3.2 ENERGY DISPERSIVE SPECTROSCOPY, SEM IMAGE SAMPLE NO.6 BEFORE AND AFTER CO₂ ABSORPTION

The elemental composition of soda lime is shown in Table

4. The main metal oxides determined by the EDS were found calcium 51.90%, sodium 3.36%, carbon 4.94% and oxygen 39.80% before CO₂ absorption and Ca (55.40%), Na (1.37%), C (6.27%) and O (36.96%) after CO₂ absorption and is shown in Fig.9. The changes in the material before and after CO₂ absorption is also clear from SEM studies and is shown in Fig.8.



Lsec: 30.0 Cnts 0.000 keV Det: Octane Super Det



Lsec: 30.0 Cnts 0.000 keV Det: Octane Super Det

Fig.9 EDS of sample no.6 element compositions before and after CO₂ absorption

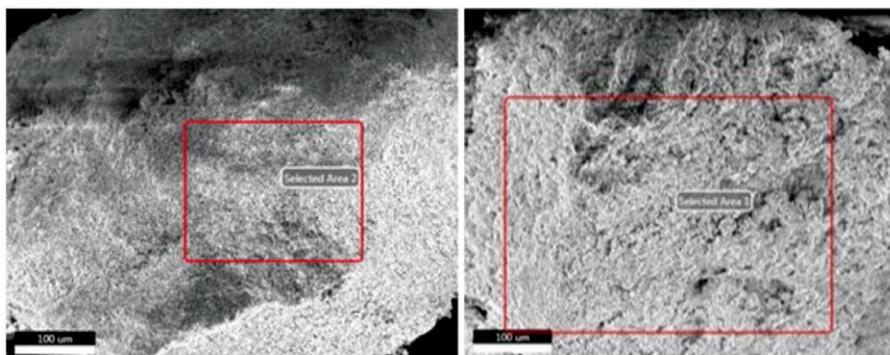


Fig.8 SEM (Scanning Electron Image) Sample no.6 before and after CO₂ absorption

3.3. X-RAY DIFFRACTION OF THE SAMPLE NO.5 BEFORE AND AFTER CO₂ ABSORPTION

Fig.10 shows that XRD spectrum of the sample with the presence of major peaks of Ca(OH)₂ and NaOH phases. However, the XRD spectrum also shows a very broad diffraction pattern where the presence of low-intensity peaks identified are clearly seen. Sample No.5 is associated with the presence of 7.15% amount of calcite

TABLE 4: EDS OF SAMPLE NO.5 AND 6 BEFORE AND AFTER CO₂ ABSORPTION

Sample	After CO ₂ absorption				Before CO ₂ absorption			
	Element	Weight %	Atomic %	Ratio	Element	Weight %	Atomic %	Ratio
Sample 5	O	37.07	60.43	0.1556	O	30.32	51.08	0.0852
	Na	1.22	1.38	0.0082	Na	0.12	0.14	0.0008
	Al	14.35	13.87	0.1238	Al	4.15	4.14	0.0348
	Si	1.54	1.43	0.0137	Si	2.25	2.16	0.0205
	Au	13.36	1.77	0.1000	Ca	63.16	42.48	0.5954
	Ca	32.46	21.12	0.3069				
Sample 6	C	6.27	12.21	0.0465	C	4.94	9.47	0.0355
	O	36.96	54.05	0.1104	O	39.80	57.32	0.1255
	Na	1.37	1.40	0.0084	Na	3.36	3.37	0.0205
	Ca	55.40	32.34	0.5102	Ca	51.90	29.84	0.4759

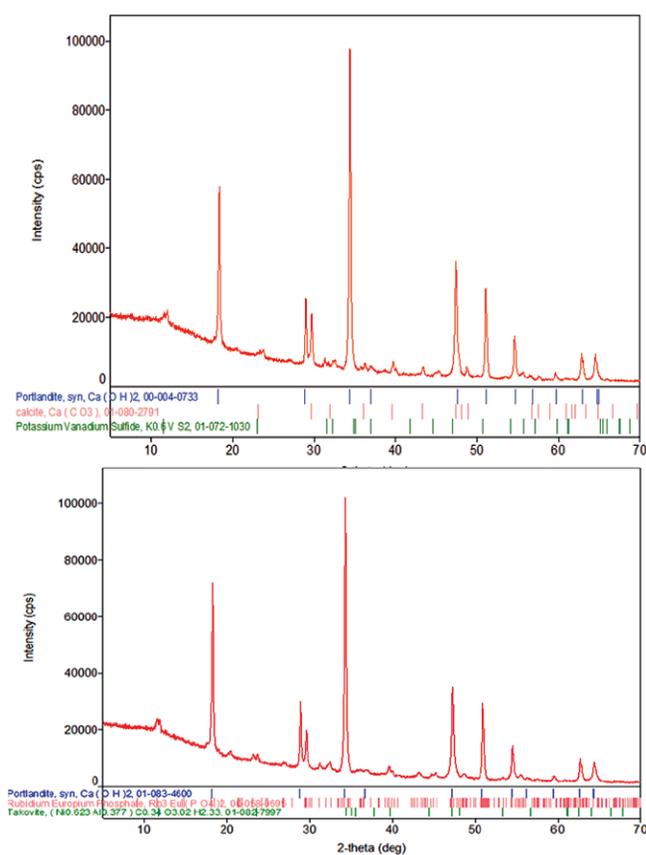


Fig.10 XRD of the sample No.5 before and after CO₂ absorption

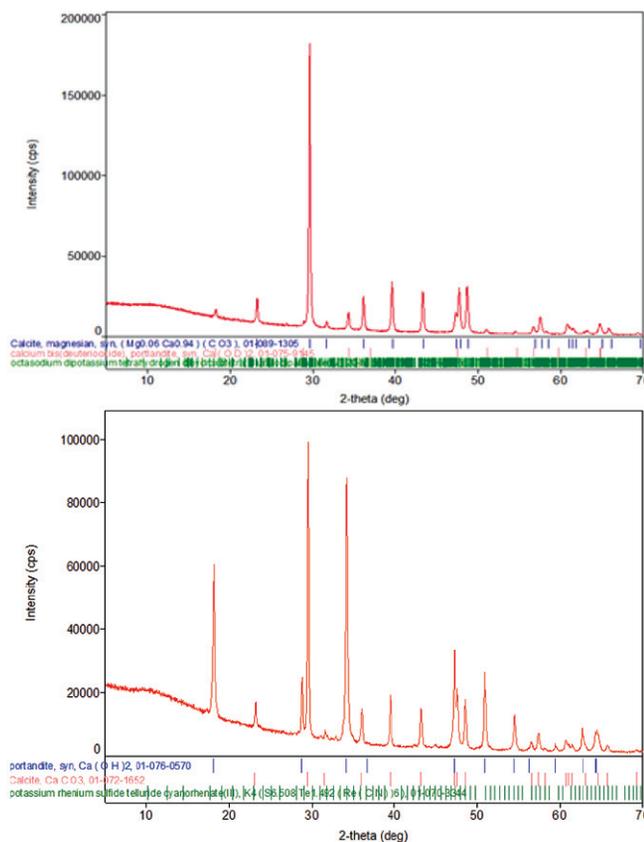


Fig.11 XRD spectra of the sample No.6 before and after CO₂ absorption

CaCO₃, 89.6% Portlandite Ca(OH)₂ and 3.24% potassium vanadium sulfide is envisaged XRD in peak and 54.8% Portlandite Ca(OH)₂, 41.2% Rubidium Europium phosphate and 4.0% Takovite [Ni₆Al₂(OH) 16(CO₃) 0.75 (OH) 0.25.4 (H₂O)] found after CO₂ absorption shown in Table 5.

3.4 X-RAY DIFFRACTION OF THE SAMPLE NO.6 BEFORE AND AFTER CO₂ ABSORPTION

Fig.11 shows that the XRD spectrum of the sample with the presence of major peaks of Ca(OH)₂ phases and other

compounds. Sample No.6 is associated with the presence of 49.6% amount of calcite (CaCO₃), 48.5% Portlandite Ca(OH)₂ and 1.8% potassium rhenium sulfide telluride cyanorhenate reaction are envisaged XRD in peaks and 89.4% calcite by XRD after CO₂ absorption shown in Table 5.

3.5 X-RAY DIFFRACTION OF UK PROTOSORB SODA LIME BEFORE CO₂ ABSORPTION

Fig.12 shows that XRD spectrum of the sample with the presence of major peaks of Ca(OH)₂ and NaOH phases.

TABLE 5: XRD OF THE SAMPLE NOS.5 AND 6 BEFORE AND AFTER CO₂ ABSORPTION

Sample No.	Before CO ₂ absorption		After CO ₂ absorption	
	Content (%)	Phase name	Content (%)	Phase name
Sample No. 5	Portlandite [Ca(OH) ₂]	89.6(7)[Peak.8]	Portlandite [Ca(OH) ₂]	54.8(5) [Peak.7]
	Calcite	7.15(8)[Peak.5]	Rubidium Europium phosphate	41.2(4) [Peak.8]
	Potassium vanadium sulfide	3.24(16)[Peak.1]	Takovite	4.0(6) [Peak.5]
Sample No. 6	Portlandite [Ca(OH) ₂]	48.5(4)[Peak.2]	Calcite, magnesian	89.4(5) [Peak.13]
	Calcite	49.6(3)[Peak.5]	Calcium bi's (deuteriooxide), Portlandite	3.38(16) [Peak.2]
	Potassium rhenium sulfide Telluride cyanoacetate (III)	1.84(14) [Peak.1]	Octasodiumdipotassium tetra hydrogen dihydroxo tetra telluratodipalladate (IV) 20-hydrate	7.2(5)[Peak.1]
UK Protosorb	Aluminum Niobium Vanadium		5.22(5) [Peak.15]	
	Portandite (Ca(OH) ₂)		94.8(5) [Peak.5]	

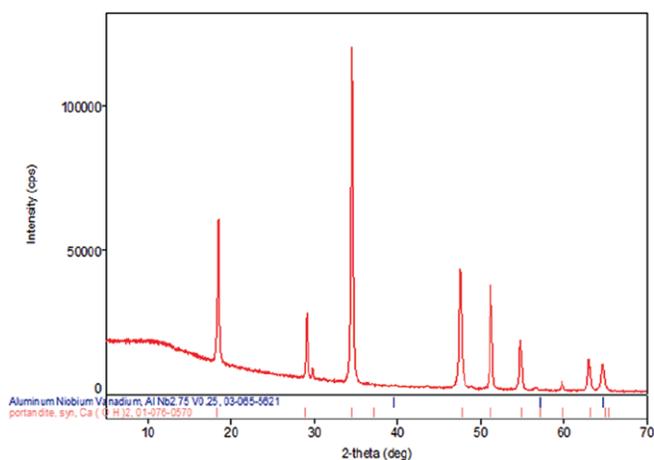


Fig.12 XRD spectrum of the UK Protosorb soda lime before CO₂ absorption

However, the XRD spectrum also shows a very broad diffraction pattern where the presence of low-intensity peaks identified clearly seen. British soda lime sample is associated with the presence of 94.8% Portlandite Ca(OH)₂ and 5.22% aluminum niobium Vanadium is envisaged XRD in peak and shown in Table 5.

3.6 COMPARATIVE STUDY OF INDIAN SODA LIME WITH UK PROTOSORB SODA LIME

The Indian soda lime moisture content varies from 11.6-18.3%. Particle size (~600µm) varies from 0.2-1.9% whereas hardness of the granules varies from 70.0-96.1% and carbon dioxide absorption time varies from 35-57 minutes but UK Protosorb soda lime moisture range is found between 16.4 and 16.5%. Particle size (~600µm) is found 0.7% whereas hardness is found 79.0% and above all activity time is found 135 minutes which are greater than Indian soda lime samples. The XRD of Indian soda lime shows that it contains calcium hydroxide [Ca(OH)₂] 48.5%, calcite 49.6% but UK Protosorb soda lime XRD result show that it contains calcium hydroxide [Ca(OH)₂] 94.8% and aluminum, niobium and vantium 5.22%

which are greater than indian soda lime.

4.0 Conclusions

Characterization of seven samples of soda lime have been done with respect to moisture, size grading, hardness and activity as per IS:5321,1969 to see its suitability for using as CO₂ absorbent in breathing apparatus which is carried out by rescue operators in case of any mine accidents. It is found that out of seven samples only four samples (1, 4, 6, 7, 8) fulfilled the criteria of activity test (55, 57, 135 minutes) and moisture and hardness but out of these only sample no.7 is not meeting the size requirement of 600µm whose permissible value is 1%. Thus it is inferred that sample no.1, 4, 6, 8 are meeting all the IS standard requirement because size of fine dusts play an important role in breathing apparatus. Whereas 2,3,5,7 are not meeting IS: 5321/1969 requirements. Out of the two sets of samples (5&6) XRD, EDS and SEM studies have been carried out to see the differences in the structure and chemical composition of the compounds. XRD sample no.5 which dose not fulfill all the criteria of IS standard shows that it contains Portlandite 89.6%, calcite 7.15% and potassium vanadium sulfide 3.24% before CO₂ absorption and Portlandite 54.8%, Rubidium Europium 41.2% and Takovite 4.0% after CO₂ absorption. But XRD of sample no.6 which fulfilled all the criteria of IS Standard Portlandite 48%, calcite 49.6% and potassium rhenium sulfide telluride 1.84% before CO₂ absorption and calcite 89.4%, calcium bi's (deuteriooxide) 3.38% and octasodium-dipotassium 7.2% after CO₂ absorption. Therefore sample no.6 is better than sample no. 5 compared but XRD of sample no. 8 is British soda lime sample is associated with the presence of 94.8% Portlandite Ca(OH)₂ and 5.22% aluminum niobium vanadium is found whose activity result 135 minute more than all samples are better. EDS of sample no. 5 which dose not fulfill the all criteria of IS standard shows that it contains oxygen 30.32%, sodium 0.12%, aluminum 4.15%, silicon 2.25% and calcium 63.16% before CO₂ absorption reaction and oxygen 37.07%, sodium

1.22%, aluminum 14.35%, silicon 1.54% Au 13.36%, calcium 32.46% after CO₂ absorption. But EDS of sample no. 6 which fulfilled the all criteria of IS Standard shows that it contains carbon 4.94%, oxygen 39.80% sodium 3.36% and calcium 51.90% before CO₂ absorption and carbon 6.27%, oxygen 36.96%, sodium 1.37% and calcium 55.40% after CO₂ absorption. This observable fact is explained through the information that calcium hydroxide, the more vigorous and soluble alkali in the lime, preferentially form carbonate. The results obtained from our works show that quality of the commercial soda-lime product used as a CO₂ absorbent material follows with the demand set by many industries for use in coal mines, navy and space capsules. This study also shows that proper storing will extend the sample shelf life by accurately amount of moisture, size grading and dust particle of soda lime.

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