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Assessment of SO_2 Emissions from Cement Industries Utilizing Limestone with High Pyritic Sulfur Content: Case Study of Cement Plants in the Jaintia Hills District, Meghalaya, India

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ABSTRACT

In properly operated Cement Plants, SO_2 emissions are mostly caused by pyritic sulfur (sulfides) in the used limestones, accounting for approximately 85% of the raw mill in the plant. However, the pyritic sulfur content in limestones of the Jaintia Hills district of Meghalaya and their influence on the SO_2 Emission from cement industries of Meghalaya have not been studied so far. The current study is conducted to perform an in-depth investigation of pyritic sulfur content in limestone reserves used by Meghalaya Cement Industries to assess the SO_2 emission in the cement industries using high pyritic sulfur limestones and review the existing technology for the recommendation of the most suitable technology to minimize the SO_2 Emissions. Random testing of collected limestone samples from various locations of Captive Mining sites in Cement Industries is performed to assess average pyritic sulfur concentration along different mining benches. Pyritic Sulfur Content (wt.%) in collected limestones varies from 0.15% to 3.5%. Polynomial Regression Analysis shows that Avg. SO_2 Emission(Y) from Klin Stack can be represented as a function of pyritic sulfur content (X) (wt.%) of used limestones in the process: $Y = 273.7X^2 + 21.46X + 422.76$. Based on the pyritic sulfur content in limestones, it is observed that “the more the Pyritic Sulfur content is, Darker the Limestone Samples are.” Hence, A Colour Scale has been prepared to visualize higher pyritic sulfur content in limestones. For longer-term sustainability, installing a Flue-Gas Desulfurization (FGD) unit at the kiln stack outlet may be included in the manufacturing process of cement plants to reduce the SO_2 Emissions from Stack.

INTRODUCTION

India is a multicultural nation with potentially valuable mineral resources. India produces over 90 different minerals, according to the Indian Mineral Yearbook Report (2013). There are 53 non-metallic minerals, 11 metallic minerals, 4 fuel minerals, and 23 minor minerals (building and other materials). This suggests that India’s mining sector is vital to economic growth. Cement is a vital building material made from limestone, a non-metallic mineral necessary raw material. India has 184,935 million tonnes of estimated limestone resources across all types and grades. 14,926 million tonnes (or 8%) are classified as reserves, while 170,009 million tonnes (or 92%) are classified as residual resources. About 9% of India’s entire limestone resources come from the northeastern states of India (Lamare & Singh 2016).

Meghalaya, one of the eight states that comprise India’s Northeastern Region (NER), is located between latitudes 25°02’E and 26°07’N and longitudes 89°49’E and 92°50’E. The Khasi, Jaintia, and Garo, are three hilly regions. There are currently 11 districts in the state. Limestone is Meghalaya’s most frequently found and exploited mineral, second only to coal. The area’s limestone rocks are found in various grades and sizes. The state’s southern border stretches for around 200 Km from the east Jaintia Hills to the west Garo Hills. According to the Directorate of Mineral Resources, Government of Meghalaya (2022), the total deposit of limestone in the state is 15,100 MT, with 9,515 MT, 41,599 MT, and 3,986 MT being the proved, indicated & inferred deposits. Tripathi et al. (1996) claimed that there is reportedly a maximum limestone reserve in Jaintia Hills (~55%). Limestone is mostly found around the southern edge of the Meghalaya plateau and is a part of the Khasi, Jaintia, and Garo groups of Cretaceous-Tertiary sedimentary rocks (Sarma 2003).

Although Meghalaya is very rich in high-quality limestone (CaO wt.% varies from 40%-55%) deposits, the Pyritic Sulfur content is reportedly high in the very high-quality limestone available in Meghalaya. This has led to the

Table 1: Emission limits from rotary kiln cement industries.

Industry	Parameter	Date of commissioning	Location	Concentration not to exceed in $mg.Nm^{-3}$
Cement plant (Without coprocessing) Standalone Clinker Grinding plant or Blending plant (MoEFCC 2016)	SO_2 in $mg.Nm^{-3}$	Irrespective of the date of commissioning,	Anywhere in the country	100, 700, and 1000 when pyritic sulfur in the limestone is less than 0.25%, 0.25% to 0.5%, and more than 0.5%, respectively

varying emission of SO_2 from the cement mills. The Ministry of Environment, Forests and Climate Change (MoEF&CC), Government of India, had notified the revised limits from emissions from cement industries on May 9, 2016. The new limits with respect to SO_2 emission from rotary kilns cement industries are represented as follows:

As seen from Table 1, the concentration of pyritic sulfur has been categorized into three broad categories viz less than 0.25%, 0.25% to 0.5%, and more than 0.5%, respectively. These are the general concentrations of pyritic sulfur in limestone across the world. However, the concentration is expected to be significantly higher in Meghalaya, especially in the East Jaintia Hills district. Various cement Industries in Meghalaya reported high concentrations of pyritic sulfur in the limestones of their captive mining. Therefore, compliance with the prescribed limit becomes a challenge for them. In this regard, this study is conducted to perform in-depth investigation of pyritic sulfur content in limestone reserves of Meghalaya Cement Industry, assess compliance w.r.t Regulatory Norms of SO_2 emission by cement industries using high pyritic sulfur limestones and review the existing technology for the recommendation of most suitable technology to counter-act the effect of high pyritic sulfur content in the manufacturing process, thereby reducing SO_2 Emissions to meet MoEF & CC Standards.

MATERIALS AND METHODS

Study Area: The Jaintia Hills district of Meghalaya is home to many cement factories, largely owing to the region's plentiful limestone resources. Clusters of several cement manufacturing facilities surround the NH-44 road from Shillong to Katigara (see Fig. 1 (A&B)). Based on operating status and those that use their own captive limestone mining reserve, the following cement factories have been selected for this study.

Limestone samples from random sites & benches across these cement industries were collected to analyze the raw limestones' pyritic sulfur concentration (wt.%). Limestone Samples from the crushed limestone feeding system of cement industries were also collected to check the inlet pyritic sulfur concentration of the rotary kiln. Further, compliance with the prescribed limits of SO_2 Emission was verified manually through stack monitoring during the field visits.

Cement industry-process flow diagram: A general flow diagram of the cement industry is shown in Fig. 2. All the cement industries under discussion share the same fundamental process design. Before the mixed flow is transferred to the preheater, crushed limestone from the limestone hopper and additives like clay, silica, or iron ore from the additives hopper are mixed in the raw mill section.

The material is heated, combined with coal from the coal mill in the rotary kiln, and then fed into the cement mill silo after cooling.

The main elements of a contemporary dry cement manufacturing process, including preheating, pre-calcination, burning, and clinker cooling, are shown in Fig. 2. The multistage cyclone preheater warms the raw mill materials. The raw meal is fed into the cyclone preheater at the top stage by being dispersed with the hot flue gas entering the cyclone inlet. The cold raw mill particles are separated in the cyclone following a direct heat transfer with the hot flue gas. The process is repeated until the final stage cyclone when the separated raw mill particles are put into the calciner. The raw mill particles are heated from about 100°C to 800°C after the typical counter current heat exchange through the cyclone preheater. Because of the calciner's comparatively

Table 2: Selected Cement Industries in Meghalaya for Study.

Sl. No.	Name of the Cement Manufacturing Plants	Address
1.	Dalmia Cements Bharat Limited (Adhunik Cements)	NH: 44, Thangskai, Meghalaya-793200
2.	Meghalaya Cements Limited (Topcem)	693J+FWF, Thangskai, Lumshnong, Khliehriāt, Meghalaya-793210
3.	Star Cements Limited	59HR+P8F, Lumshnong, Meghalaya 793200
4.	Hills Cements Limited	NH 44, Khliehriat, Jaintia Hills, Mynkree, Meghalaya-793200
5.	Goldstone Cements Limited	Umrasong, Meghalaya 793210
6.	Amrit Cements Limited	69MH+89G, Lumshnong, Meghalaya-793210

Fig. 1 (A): Jaintia Hills District of Meghalaya (NH-44 Road Shillong to Katigara) (Reference:www.mapsofindia.com).

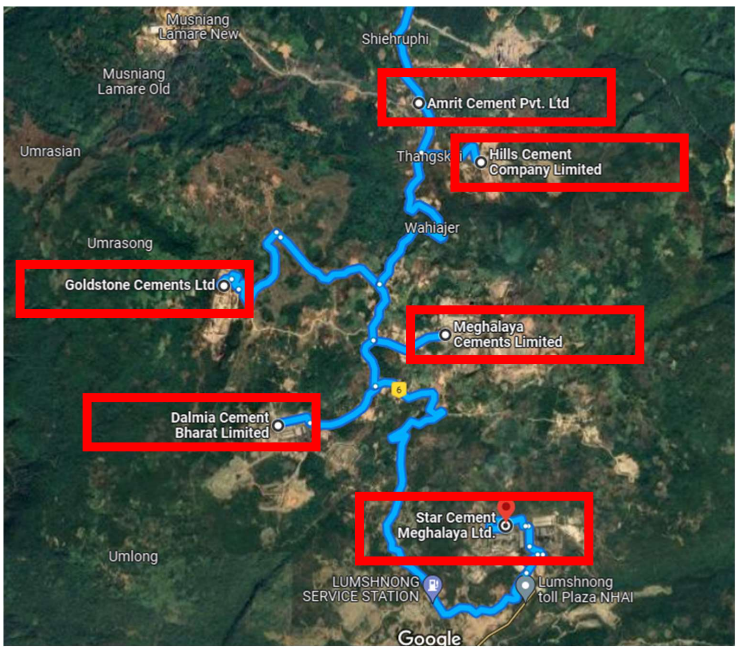

Fig. 1 (B): Distribution of Selected Cement Industries for study along NH-44 of East Jaintia Hills District (Reference: Google Maps).

high temperature of 900°C to 950°C, the limestone particles in the raw mill break down to generate CaO. The endothermic degradation of the limestone in the calciner requires additional heat, which is produced by burning fuels.

Following calcination, the calcinated raw meal is fed into the rotary kiln, where it is heated at a temperature of up to 1300°C to 1400°C to create cement clinker. Fuels are burned in the rotary kiln to provide heat. Finally, the newly formed cement clinker is cooled in a clinker cooler.

Release and Capture of SO_2 in the Process: Most SO_2 emissions occur in the preheater due to the oxidation of sulfides, primarily pyrites and organic sulfur (if any), in the raw mill. The remaining SO_2 emissions are produced in the pre-calciner and kiln by the oxidation of sulfides, primarily pyrites and organic sulfur in fuel, and the breakdown of sulfates from raw mill particles and fuel (Gossman 2011).

The raw mill, preheater, and pre-calciner all work as dry scrubbers to control SO_2 emissions because they use kiln exhaust gas for drying, heating, and calcining the raw mill particles before they reach the kiln. The particle control system in the bag-house may additionally dry scrub some SO_2 (Horkoss 2008). As a result, SO_2 is released and captured at the same time. It is worth mentioning that the raw mill and preheater have insufficient SO_2 collecting capacity due to the slow reaction rate between SO_2 and $CaCO_3$. The bulk of SO_2 may be trapped when flue gas goes through the pre-calciner because there is a lot of CaO there, and SO_2 and CaO react fast when they come into contact. Alkali and CaO in the kiln can also absorb SO_2, but during clinkerization, some amount of $CaSO_4$, the primary desulfurization product, will break down and release SO_2 again (Zhang et al. 2019).

Organic and inorganic sulfurs can be found in raw mills and fuel, primarily referring to sulfides (such as pyrite) and sulfates (gypsum). More than 80%-90% of the sulfur in

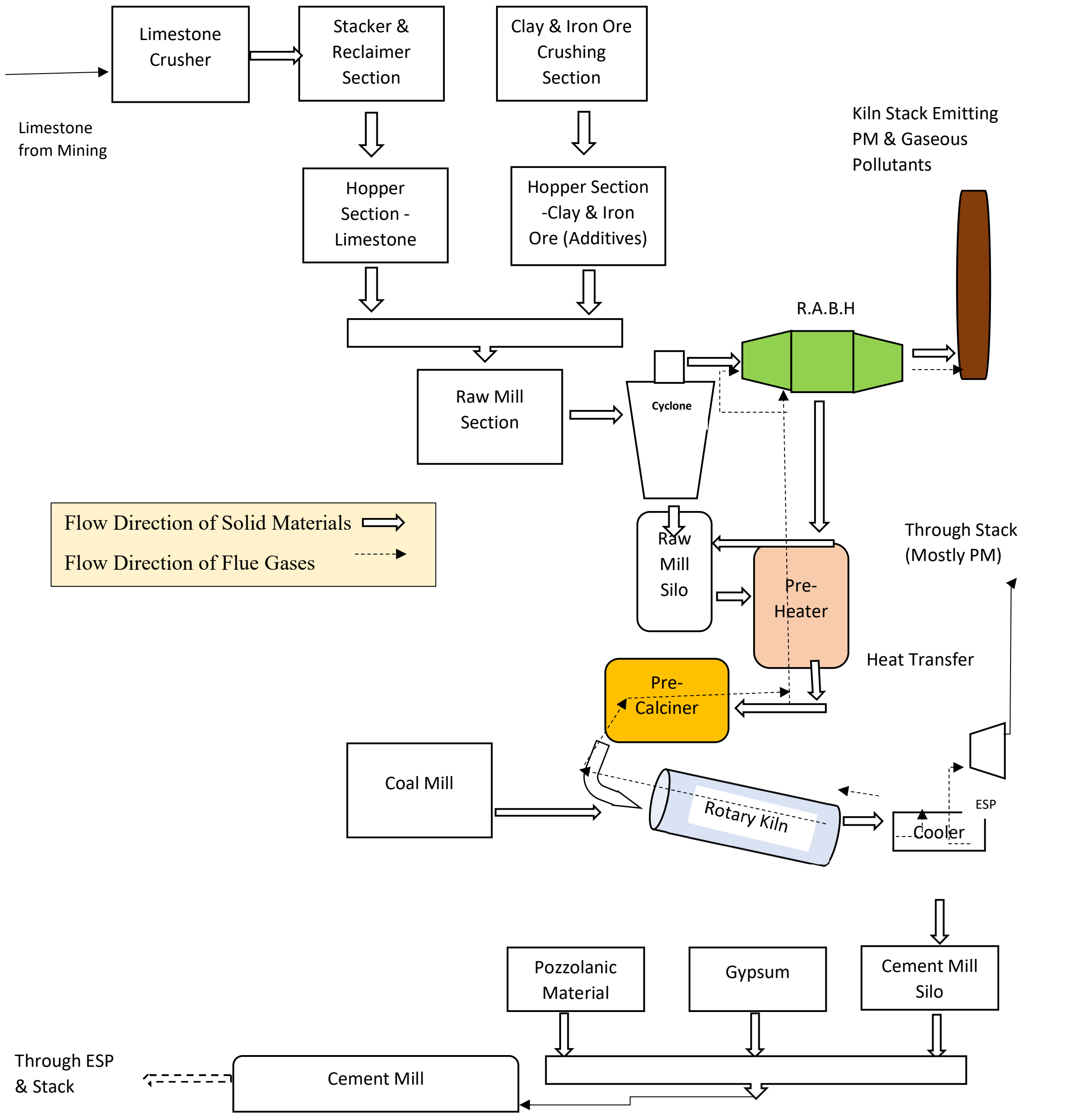

Fig. 2: Process Flow Diagram of Cement Plants.

coal is organic, with the rest primarily made up of pyrite (FeS_2), gypsum ($CaSO_4$), and a few ferric sulfates (Oliveira et al. 2011). Whether the coal is burned in the kiln or CaO may absorb the pre-calciner, the bulk of the SO2 released by the coal as the flue gas passes through the pre-calciner. On the other hand, the source of sulfur in limestone is purely inorganic: it primarily exists in the form of pyrite (Sulfide form) and gypsum/sulfate form. Because pyrite sulfur is more unstable than sulfate sulfur, pyrite sulfur would be released as SO_2 first compared with Sulfate decomposition.

Since the loading (wt.%) ratio of limestones& fuels (coal) ranges around 85:15, the minimal SO_2 Emission contributed by coal combustion in pre-calciner or kiln can be captured by CaO (forming $CaSO_4$) when the flue gas passes through the pre-calciner. Because the raw mill and preheater have a limited capacity for absorbing SO_2, any increase in the amount of pyritic sulfur in the raw limestones would eventually cause the preheater to oxidize the pyritic sulfur and release surplus SO_2, which the flue gas would then transport till stack emission. In light of this, the primary defining element for SO_2 emission from the stack associated with Kiln flue gas is the Pyritic Sulfur Content of limestones from raw limestones.

Methodology

Limestone samples were collected from different sites of mining benches (See Fig. 4) of the selected cement industries for analyzing the pyritic sulfur concentration (wt.%). Limestone Samples from the crushed limestone feeding system before the Kiln inlet were also collected from the selected plants to check the inlet pyritic sulfur concentration of the rotary kiln. The system adopted for minimizing pyritic sulfur in the kiln inlet was also deeply evaluated during field visits. Compliance with the prescribed limits of SO_2 Emission was verified manually during the field visits. The Methodology Adopted for this study is summarized in Fig. 3.

The collected samples were sent to M/s National Council for Cement and Building Materials, Haryana, India, for analysis of the pyritic sulfur content in the collected samples. Test Method *ASTM C-25: 2019* is used for reporting the

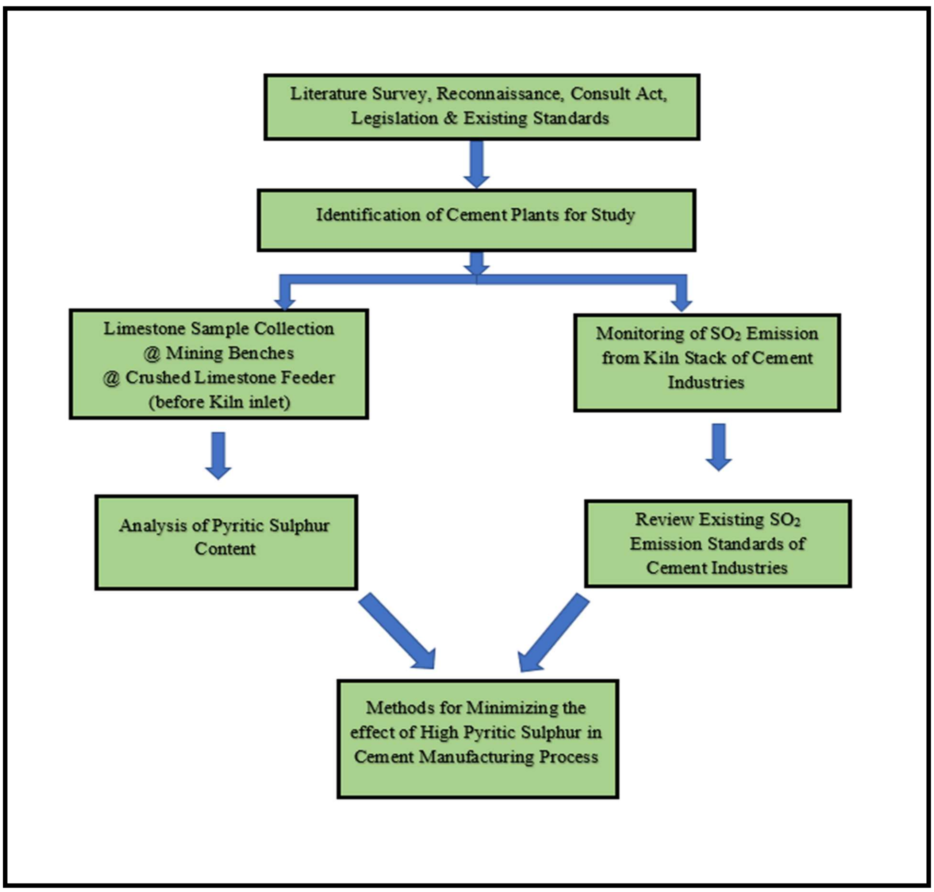

Fig. 3: Methodology of the study.

Fig. 4: Limestone mining bench @ Meghalaya Cements Ltd. (Topcem).

pyritic sulfur content (wt.%). There is no standard test method for directly determining pyritic sulfur in limestone. However, as per clause 24.1 (Scope) of ASTM C 25- 17, sulfur in limestone is chiefly, if not wholly, present as sulfide, usually as pyrite. If the total sulfur obtained in the Sodium carbonate fusion method is more than that present as soluble sulfate, the difference can be assumed to be iron disulfide." The concentration of pyritic sulfur in limestone samples is determined using this indirect technique.

RESULTS AND DISCUSSION

Comparison of pyritic sulfur content v/s SO_2 emission: Limestone samples were collected from various limestone mining areas & processing sites of the selected cement industries to analyze the pyritic sulfur content (wt.%). Fuel Samples (Coal) were also collected from several Industries to assess the contribution of pyritic sulfur content (wt.%) at the Kiln inlet.

Fig. 5 depicts the variation of pyritic sulfur content (wt.%) in limestones across different Mining Benches of Cement Industries in Meghalaya. While most mining benches have Pyritic Sulfur content (wt.%) values ranging from 0.5% to 0.7%, Mining benches of Dalmia Cements show huge variation in the values of Pyritic Sulfur. Among the Four Benches (4) of Dalmia Cements Ltd., the limestones of the two benches have high pyritic sulfur (wt.%), i.e., 1.5% and 3.5%, respectively. Even limestone samples collected from Weigh Feeder before Kiln inlet is found to be higher (0.65%) for Dalmia Cements Ltd. (Fig. 6). High Pyritic Sulfur Content (wt.%) in limestones of mining benches at Dalmia Cements Ltd also contributes to the plant's relatively greater SO_2 Emission (941.3 $\mu g.Nm^{3-}$) from Kiln Stack than others (Refer Fig. 7).

An attempt has been made to correlate the Average pyritic Sulfur concentration (wt.%) in limestones used by the cement plants in Meghalaya with their SO_2 emission from the kiln stack. Polynomial Regression Analysis (Refer Fig. 7) shows that Avg. SO_2 Emission(Y) from Klin Stack can be represented as a function of pyritic sulfur content (X) (wt.%) used limestones in the process:

$$Y = 273.7X^2 + 21.46X + 422.76$$

Although all the cement industries under investigation are complying with the standards notified by the Ministry of Environment, Forests and Climate Change, Government of India (GoI) (Standards can be referred from Table 1), SO_2 emission (941.3 $\mu g.Nm^{3-}$) from M/s Dalmia Cements Ltd was found to be high compared to other plants & the value is almost touching the Maximum limits of 1000 $\mu g.Nm^{3-}$. High pyritic sulfur content in the mining benches at Dalmia Cements Ltd is the main contributor to such a phenomenon. M/s Dalmia Cements Ltd. has adopted certain measures to counter-act the Average pyritic Sulfur concentration (wt.%) effects in their limestones. Selective mining from low pyritic sulfur Mining Benches and variation of Additives flow rate are practiced to tackle the rise in sulfur content in the feeder section & hence in SO_2 emission in the stack. The analytical Sulfur content of crushed limestones is checked hourly in the sample point of Limestone Hoppers. If the sulfur content exceeds the desired limit, the same is neutralized by the increase in additives flow rates from the additives hopper. Thus, the material in the Raw Mill, Sulfur content, is well within the desired limit. But this is a short-term preventive measure and a very tedious work.

Color scale based on pyritic sulfur content in limestones: We noticed color variation in crushed limestone samples collected from different mining benches. Increasing pyritic sulfur content in limestone samples has resulted in a color intensification trend (Fig. 8).

Notably, dark grey limestones have a higher pyritic sulfur than light grey limestones. In dark grey or dark

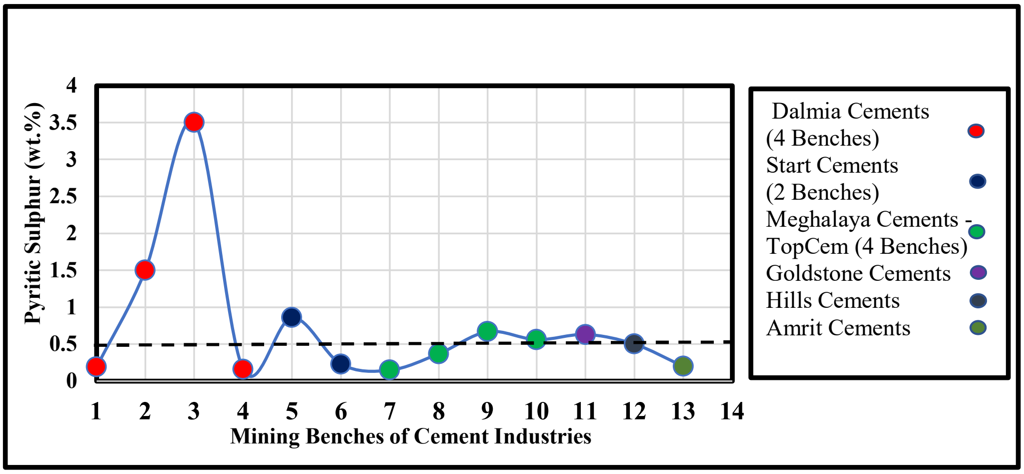

Fig. 5: Pyritic Sulphur Content in Limestones accross Mining Benches.

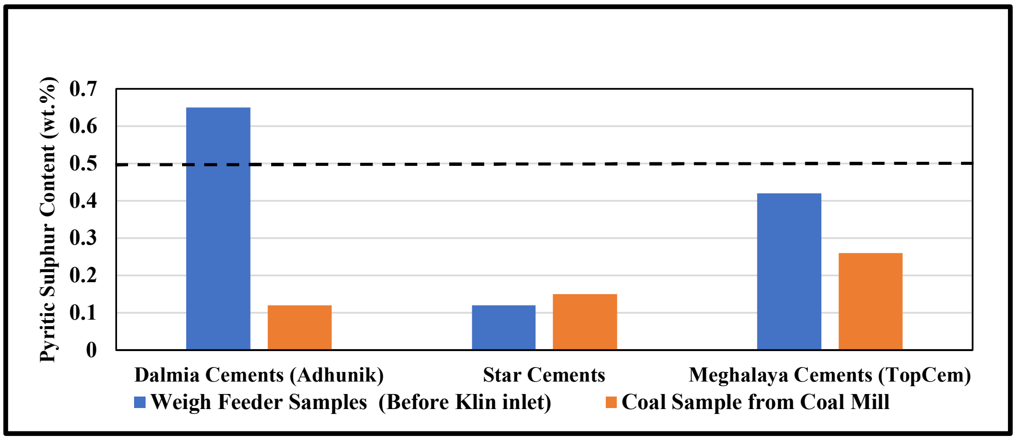

Fig. 6: Pyritic Sulphur Content in Limestone and Fuel Samples collected from Processing Sites.

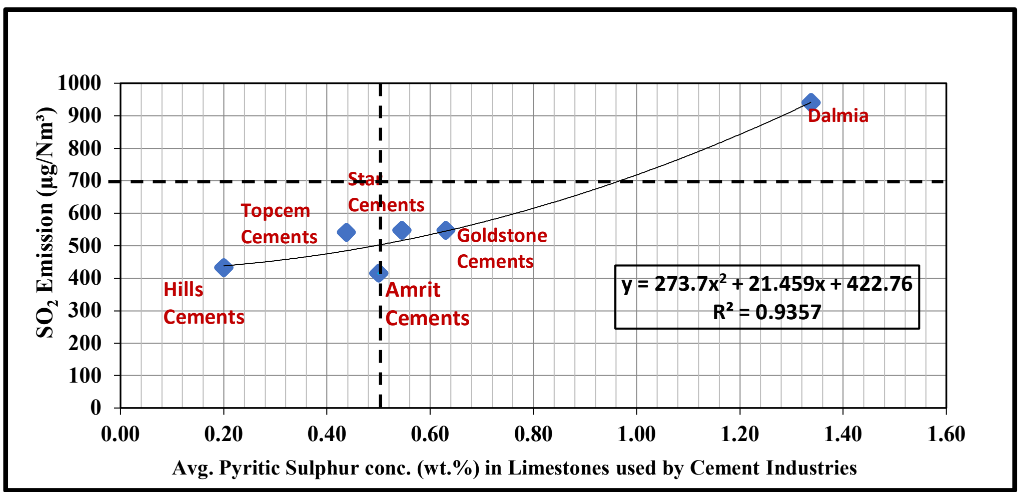

Fig. 7: Avg. SO_2 emission from klin stack v/s Avg. pyritic sulphur content in used limestones (Stack emission as on Oct. 2022).

brown limestones, bulk pyrites (FeS_2) are found layered and dispersed with impurities (In a reductive environment, metamorphic or sedimentary rocks typically develop). On the contrary, light yellow or light grey limestones, often calcite sedimentary rocks with trivalent iron oxides or hydroxides, hardly ever contain bulk pyrite. It is commonly known that trivalent iron oxides or hydroxides only form in oxidizing atmospheres, whereas pyrite only occurs in reducing atmospheres. As a result, light yellow or light grey limestones rarely contain pyrite and have substantially lower sulfur contents than dark grey limestones (Zhang et al. 2019).

Based on the color of the collected limestone samples and their pyritic sulfur content, a Colour Scale has been prepared to visualize higher pyritic sulfur content in limestones. (Fig.9) Colour Scale Can be easily used in fields to quickly understand pyritic sulfur in the limestone mining bench.

Measures for Minimization of Effect from High Pyritic Sulfur in Cement Manufacturing Process:

Pyritic sulfur in limestones is highly undesirable in the cement industry. It not only sets the stage for the significant SO_2 emissions from the kiln, but it also causes the production

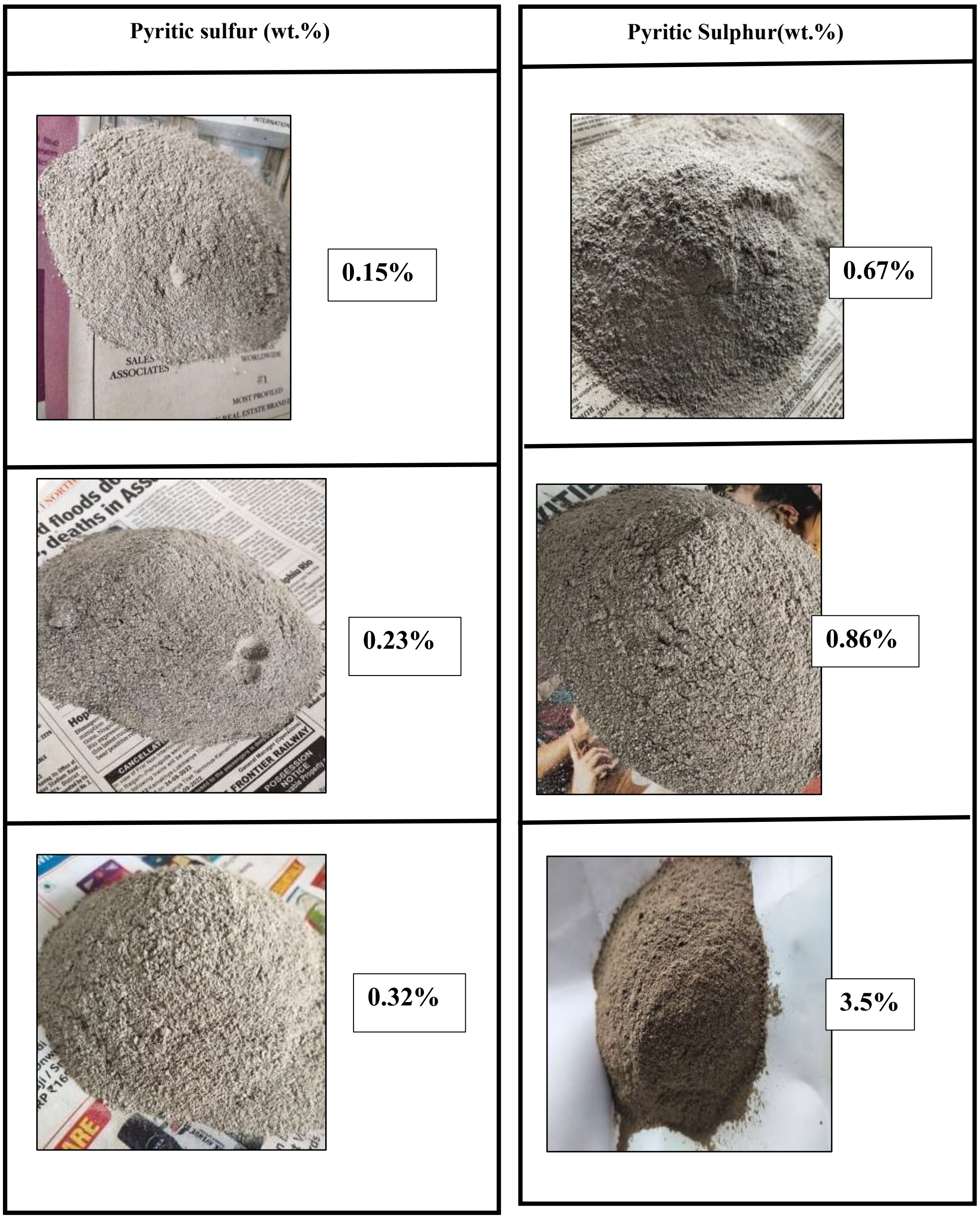

Fig. 8: Colour Intensification Trend in Collected Limestones.

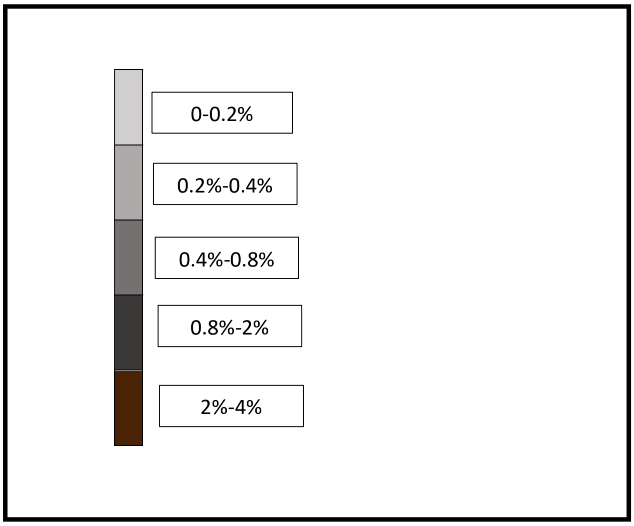

Fig. 9: Colour Scale for identification of pyritic sulphur (wt.%).

of golden rings inside the kiln skin, raising the system's maintenance costs. Several Steps are taken to minimize the effect of the same. We have made an effort to talk about a handful that emerged from the study and fieldwork.

A. Short-Term Measures

- **Selective mining and variation of additives flow rate** can be used to tackle the rise in sulfur content in the feeder section & and, hence, in SO_2 emission in the stack. As opposed to mining benches with high pyritic sulfur content, mining benches with low pyritic sulfur content can supply raw limestones in cement plants. After a proper sulfur distribution study in mining benches, a Minimal proportion of limestones from Mining benches with high pyritic sulfur content may also be blended with limestones from mining benches with low pyritic sulfur content, keeping an eye on the pyritic sulfur content in the weigh feeder section. A case Study of Dalmia Cements Ltd. may depict a proper understanding of the blending ratios to get the required pyritic sulfur content in the Weigh Feeder (Refer to Table 3).

It is very much evident from Table 3 that About 80%-90% of the blended limestones should be from the mining benches with low pyritic sulfur content (wt.%) to get the desired kiln inlet pyritic sulfur value of 0.41%-0.64%. Moreover, the concerned ministry/department must investigate the distribution of pyritic sulfur content in captive mining benches of cement plants before giving any Environmental Clearance or Consent.

Table 3: Blending Proportions from Various Mining Benches of Dalmia Cements Ltd.

	Mining Benches	Pyritic Sulphur Content(wt.%)	Blending Proportions	Pyritic Sulphur in BlendedLimestones
Case-1	1	0.19%	0.4	0.64%
	2	1.50%	0.1	
	3	3.50%	0.1	
	4	0.16%	0.4	
Case-2	1	0.19%	0.2	1.57%
	2	1.50%	0.3	
	3	3.50%	0.3	
	4	0.16%	0.2	
Case-3	1	0.19%	0.45	0.41%
	2	1.50%	0.05	
	3	3.50%	0.05	
	4	0.16%	0.45	

- **The lime dosing system in the preheater** removes sulfur from the stream, and the produced $CaSO_4$ (Gypsum) is removed from the outlet of the kiln.

B. Long Term Measures:

- The cement plants may install **a Flue-gas desulfurization (FGD) unit** to treat the flue gas from the kiln. Because of the acidic nature of emitted SO_2 in flue gas, the typical alkaline sorbent slurries or other materials like $CaCO_3$ (limestone) slurry or Ca $(OH)_2$ (hydrated lime) slurry are used to remove the SO_2. The reaction produces $CaSO_3$ (calcium sulfite). Further, $CaSO_3$ (calcium sulfite) is oxidized to produce marketable $CaSO_4 \cdot 2H_2O$ (gypsum) used in wallboard and other products.

Being Capital-intensive, Industries are reluctant to adapt to this new technology. However, a proper techno-economic feasibility study may be performed before reaching any conclusion.

Being Capital-intensive in nature, Industries are reluctant to adopt this new technology. However, a proper techno-economic feasibility study may be performed before reaching any conclusion.

CONCLUSION

The Pyritic Sulfur of limestones from the majority of cement industries in Meghalaya was found to be within values ranging from 0.5% to 0.7%, except Mining benches of Dalmia Cements show huge variation in the values of Pyritic Sulfur. Among the Four Benches (4) of Dalmia Cements Ltd., the limestones of the two benches are found to have high pyritic sulfur (wt.%), i.e., 1.5% and 3.5%, respectively. Even limestone samples collected from the Weigh Feeder before the Kiln inlet are higher (0.65%) for M/s Dalmia Cements Ltd than other plants. Although SO_2 emissions from kilns from cement industries were also within the acceptable range as notified by MoEF&CC, GoI, SO_2 emission from M/s Dalmia Cements Ltd is high compared to other plants. The cement industries may use selective mining from high and low pyritic limestone mining benches to tackle the rise in sulfur content in the feeder section. Installing a Flue-Gas Desulfurization (FGD) unit at the kiln stack outlet may be included for longer-term sustainability. The pyritic sulfur content of utilized coals is found to have relatively little effect on SO_2 emissions in Meghalaya cement factories. However, SO_2 released from the alternative fuels was not considered for this study. For example, co-processing high sulfur wastes, especially municipal solid waste, waste tires, etc., may also create high SO_2 emissions in blocks of cement industries of Meghalaya. Therefore, the effects of sulfur characteristics of alternative fuels and treatment techniques for wastes containing sulfur should be further studied in the future.

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