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Effect of Calcination Condition and Cooling Method on Reactivity of Lime Sludge

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Abstract

In this Experimental Study, Raw Lime sludge from the Paper industry was characterized by XRD and TGA techniques. Lime Sludge was calcined for a temperature range of 550–750°C for 2 h and cooled by air cooling. The effect of calcination temperature on the reactivity of resultant Sludge was investigated by various indirect and direct methods of pozzolanic activity assessment such as the Strength Activity Test (SAI), Lime reactivity test, XRD, TGA, and API. It was concluded that the calcination of lime sludge affects the microstructure and pozzolanic reactivity of the resultant Sludge. For Calcination temperatures lower than 700°C, Calcinations of $CaCO_3$ are not complete. Whereas at higher temperatures, the crystallization of amorphous content was enhanced. It is observed that calcinations of Sludge at a temperature of 750°C for 2h and slow cooling condition increases the pozzolanic reactivity of Sludge. Results showed that compared to other pozzolanic admixtures such as FA or BFS, CPS (Calcined Paper Sludge) had superior pozzolanic activity. With a calcination temperature of 750°C, a calcination time of 2 hours, and a slow cooling method, their pozzolanic activity was exceptionally good. At 28 days old, the SAI (percent) of mortar with CPS produced under ideal circumstances was 94%, comparable with the fly ash's quality (Class F).

Keywords: Lime Sludge, Calcination, Calcined Clay, SAI, XRD, TGA, Lime Reactivity Test

INTRODUCTION

Urbanization and rapid population growth, especially in developing countries, escalate housing and infrastructure demand. It is at a much faster rate that is ever experienced. Annual global cement production has reached 2.8 billion tons and is expected to reach around 4 billion tons per year in the year 2050. The epicenter of these growths is mainly china and India., with other developing regions like the Middle East and Northern Africa [1, 2, 3].

The process of cement manufacturing is highly carbon and energy-intensive. It is projected that in the production of 1 ton of Portland cement generates 730–990 kg of CO_2, which is responsible for about 8% of total CO_2 Production [4, 5]. So in recent years, the focus of the researcher shifted towards the production of new binders, which are less energy-intensive. Reduction in CO_2 emission can be accomplished by partially replaced in binder by supplementary cementitious materials (SCMs), which are mostly derived as unwanted by-products [1, 3, 5] from other industries. These byproduct is viable only if they perform similarly to or better than concretes manufactured from OPC, since these product are industrial waste so skillful utilization of these product reduce the cost of production significantly and also performance can be enhance in case of some SCM. In recent years, there are lots of attempts have been made to incorporate

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alternative materials, but its potential for further usage still exists. Subsequently, due to limitations of traditional cement constituent's material availability. Widely used alternate Materials, such as fly ash, blast furnace slags, and other natural pozzolans, are available in much less quantity compare to worldwide demand of ordinary Portland cement (OPC) [4, 6, 7]. So, research interest has been shifted towards readily available sources of SCM's such as clays and Sludge from various industries such as paper mill, textile, water treatment, sewage sludge [6, 8].

Pulp and paper industries produced a huge amount of lime sludge due. [9, 10, 11]. In India, During the white liquor recovery process, the paper companies produce about 4.9 million tons of lime sludge annually [12, 13, 14]. Because it is frequently dumped straight into landfills, lime sludge contributes to both anthropogenic and natural environmental impacts. this causes issues with the environment such carbon breakdown, microbial activity, leaching, and ion exchange processes. This led to the contamination of the surface, groundwater, and soil. It is essential to use this waste material in deserving applications like building material due to its risk to the environment [15, 16]. The major component in Lime sludge is Calcium Carbonate, while Silica and Alumina also present with small proportion [11, 17]. The two primary crystalline phases in lime sludge are kaolinite ($Al_2O_3.2SiO_2.2H_2O$, 10–20%) and calcite ($CaCO_3$, 60–90%). Reactive lime (CaO) and Metakaolin ($Al_2O_3.2SiO_2$) were produced during the thermal treatment of lime sludge, which accounts for their high reactivity when used as additional materials in cement [17, 18, 19].

MATERIALS AND METHODS

Lime Sludge

Waste produced by industries such as paper, acetylene, sugar, fertiliser, sodium chromate, soda ash, and water softening containing mainly $CaCO_3$ is known as lime sludge or Lime Mud. About 13 million tons of paper, paperboard, and newsprint produced by various Pulp and paper industries in India per annum, which account for about 3.18% of the total production (408 million tons per annum) for the world [9, 10, 19]. Paper production produces a variety of trash. For every tonne of paper produced, a pulp and paper mill generates 1.63 tonnes of lime sludge. Being the most polluting industries is a result of the vast amounts of lime sludge production [14, 15, 19].

According to the studies, cement clinker can be produced using up to 74 percent (dry basis) of the lime waste from the pulp and paper industry sector. Additionally, about 30% of dry lime sludge can be used in the manufacture of masonry cement. Because Sludge contains more calcium than other types of Sludge, it can be utilised up to 30% in cement clinker production, as opposed to only 10% for other types of Sludge [20].

Characteristics of Lime Sludge

Lime sludge is made up of very small precipitated $CaCO_3$ particles and leftover, unsettled green liquor clarifier dregs. Table 1 displays the typical Physio-Chemical characteristics of waste Lime Sludge. The Main mineralogical component of Lime Sludge is Calcite, Quartz, Hemihydrate [19, 21]. The Lime sludge

Used in this study is procured from 'BILT, Paper Industry Yamunanagar'. Figure 1 depict the Ternary Diagram of Different Waste material with respect to their composition. XRD of Raw sludge with heighted peaks of contains mineral like calcite, talc, Kaonite as main constituents in Figure 2. SEM image of Raw Lime Sludge and % weight of different Component is shown in Figure 3. Differential thermal analysis (DTA)/thermogravimetry (TG) was conducted in the temperature range of 20–1000 _C at a heating rate of 20 _C/min in flowing air Figure 4.

Cement and Aggregate

Ordinary Portland Cement (OPC), as described in IS 8112:1989 [22], was utilised for the SAI test of mortar containing Calcined Paper Sludge. Table 4 displayed the cement's chemical attributes. Additionally, 0.19 to 0.32 mm-sized domestic quartz sand having density of 2.67 g/cm3 is also used.

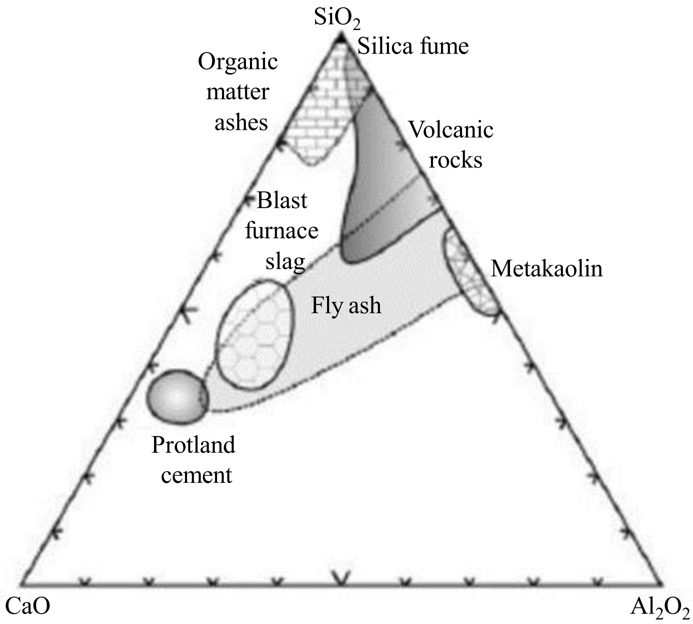


Figure 1. Ternary diagram showing position of different waste material [11].

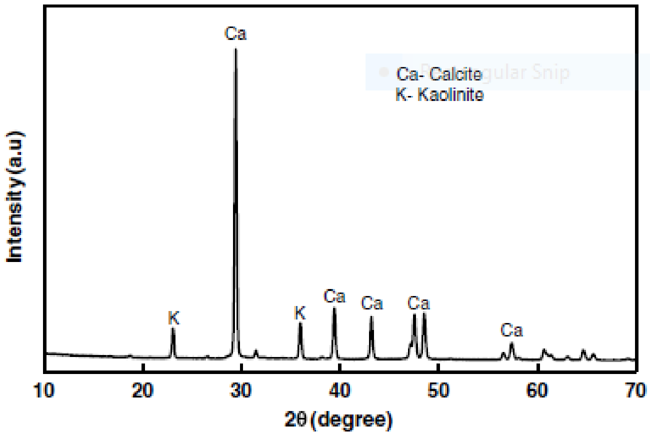


Figure 2. XRD of Raw lime sludge [6].

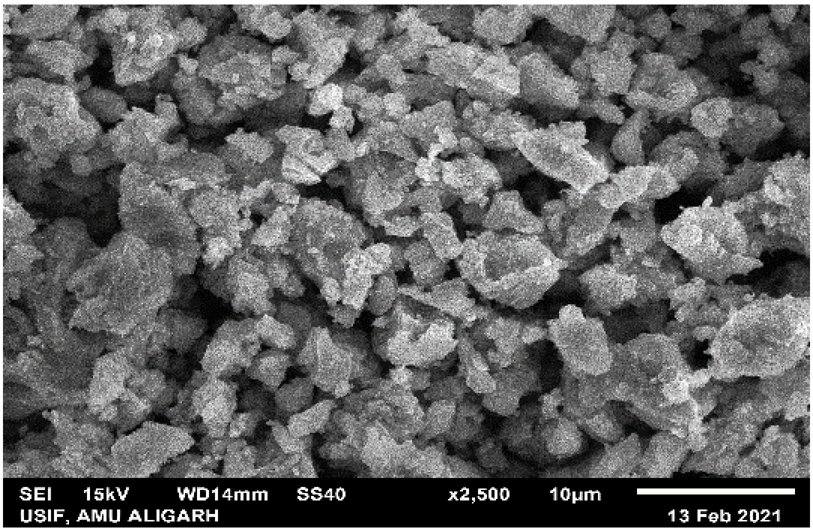


Figure 3. SEM image of raw lime.

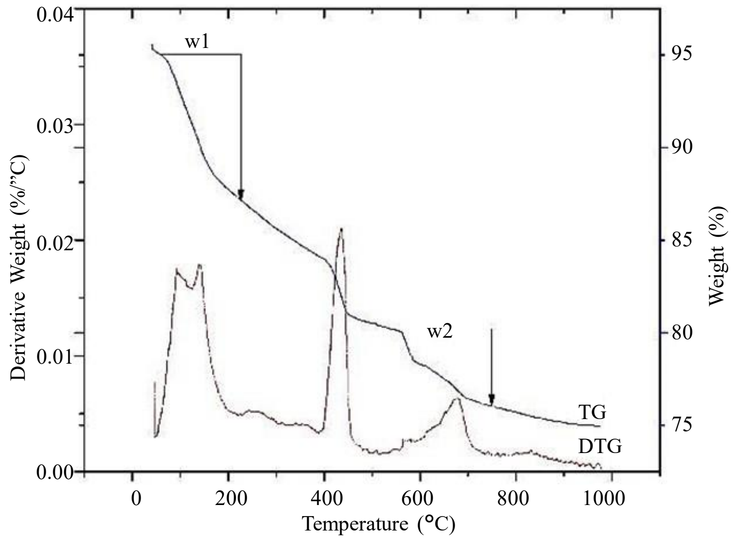


Figure 4. DTA/TGA curve of lime sludge.

Table 1. %Weight of different component in raw lime sludge.

Element	Weight %	Atomic weight%
O	58.79	75.99
Al	1.71	1.31
Si	9.72	7.16
S	2.98	1.92
Ca	25.35	13.08
Fe	1.45	0.54

Calcined Clay

Characteristics of Metakaolin

The mineralogical composition of Metakaolin as seen from XRD SEM (Figures 5, 6) presented in Table 2. MK generally contains SiO_2 (50%–55%) and Al_2O_3 (40%–45%) as found by SEM (Figure 6) and quoted by other authors as well [18, 21]. In lower concentrations, other oxides like TiO_2, Fe_2O_3, MgO, and CaO are also present. It demonstrates the existence of quartz as the primary mineral and the amorphous Alumino-silicate phase, which is produced by high-temperature interactions between SiO_2 and Al_2O_3 as well as the fluxing of oxide impurities and oxides. The XRD of calcined clay shows the presence of Quartz and Montmorillonite as a significant constituent [23, 24, 25]. With the availability of extra Ca^+ ions Metakaolin undergoes a pozzolanic reaction to form CSH when employed in concrete, which improves the microstructure of the hydrated cement paste. The calcined clay for present study was procured from' TARA, Development Alternative' Qutub Instutional Area, New Delhi.

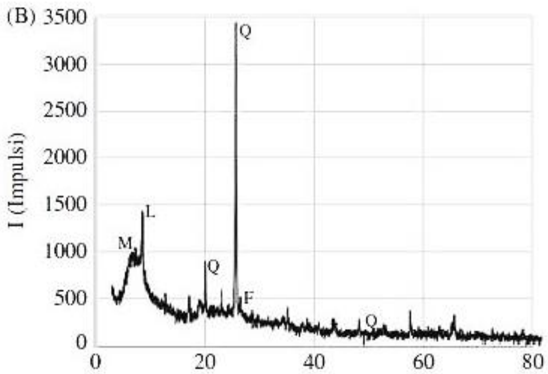


Figure 5. XRD pattern of calcined clay [23].

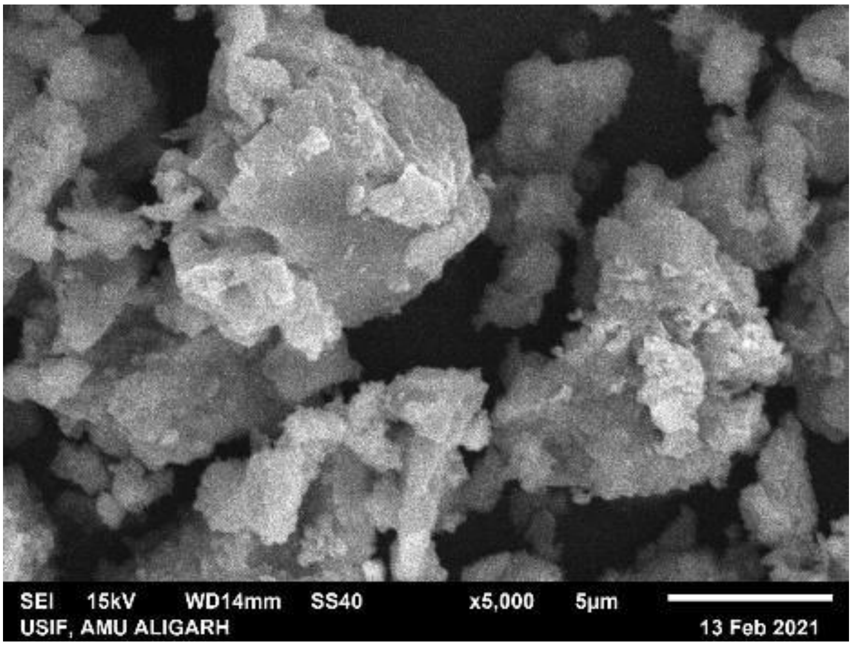


Figure 6. SEM image of metakaolin.

Table 2. %Weight of different component in metakaolin.

Element	Weight %	Atomic weight%
O	54.89	67.6
Na	1.18	1.01
Al	21.76	15.89
Si	21.94	15.39
Ca	0.24	0.12

Table 3 Experiment factors.

Factors	Limits
Temperature	550–800°C
Cooling Method	Slow cooling in Furnace

Table 4. Chemical properties of binders.

Material	Oxide Composition								
	CaO	*SiO_2*	*Al_2O_3*	*Fe_2O_3*	*MgO*	*SO_2*	*Na_2O*	*K_2O*	*TIO_2*	*LOI*
Lime Sludge	48–53	2-8	0.8–1	0.8–1.2	.2–.3	0.1–0.3	0.8–2	0.2	0.69	37–50
OPC	72.1	20.68	4.90	3.35	2.64	2.65	0.11	0.14	0.12	2.73
Calcined Clay	3.11	49.1	26.5	6.69	1.05	0.76	0.74	1.67	1.55	

Experiment Setup

In this study, optimum calcination temperature for production of a highly reactive pozzolan of paper sludge were widely investigated. Mineral components included in the Sludge's inorganic fraction, like kaolin and limestone, are receptive to such production. Then, different calcination temperatures were then used to create different calcined paper sludge (CPS) types. The Assessed Pozzolanic Activity Index and Strength Activity Index tests were used to investigate the pozzolanic activity of the CPS. [26]

Experiment Parameter consists of calcination Lime Sludge between Temperature range of 550

to 800° C and slow cooling in furnaces as listed in table 3.

METHODS

A detailed Experimental study is carried out to access the influence of calcining conditions such as temperature and cooling methods on the mineralogy and texture of calcined sludge products carried out as part of the current work, while examining the fundamental properties of paper sludge that influence pozzolanic activity. To study the effect of calcined paper sludge (CPS) on reactivity of Calcined clay paper sludge blend, paper sludge is calcined at different temperature range (500 to 750°c) in muffle furnace for 2 hours based on previous studies [6, 23, 27], then It is allowed to air cool for 24 hours

Determination of Pozzolanic Activity

Pozzolanic activity or reactivity of paper mill Sludge is determine by two ways – Direct Methods and Indirect Methods

Indirect Method

Indirect methods involve use of sophisticated techniques to quantify various chemical compounds indirectly, such as in XRD where Diffraction of various components can be represented at a particular angle and intensity, by measuring intensity at that respective angle the compound can be matched from a standard data set [23]. X-ray fluorescence is used to identify Chemical compositions of CPS samples and crystalline phases with monochromatic Cu Ka radiation. Match 3 software was used to analyses X ray Diffraction data.

In Differential thermal analysis (DTA/TG) the weight loss associated with a particular temperature range indicates the presence of that specific compound. Differential thermal analysis (DTA) and thermal gravitational (TG) was used to identify temperature effect on sample for temperature range of 20–10000 C at a heating rate of 10 C/min inflowing air [23].

Direct Method

To optimize the calcined temperature and blend proportion direct test method to used assess pozzolanic properties such as SAI and Lime Reactivity Test (IS 1727-1967).

Strength Activity Index (SAI) Test

One of the most popular methods to access pozzolanic Material's reactivity is SAI (Strength Activity Index (ASTM C618). It measures the reactivity in terms of percentage strength gain with reference to control Material, if some percentage of control material is replaced with pozzolanic Material. A material which show more than 70% strength gain with reference is said to be a pozzolanic material. The Proportion of material used for testing is shown in Table 4. Mortars tested for flow and compressive strength in accordance with ASTM C618. In SAI test, the water content is adjusted to provide the same flow value for reference as well as for blend [23, 26]

Strength Activity Index

$$SAI = A/B * 100 \ (\%) \qquad (1)$$

Where

A. Compressive strength of the test mortar (MPa) at 28 days
B. Compressive strength of the control mortar (MPa) at 28 days.

Lime Reactivity Test

In this test, mortar cubes made with Standard Lime calcium hydroxide of purity > 92% is replaced with Lime Sludge of different Calcination Temperature and SCM Metakaolin. The Lime to SCM ratio by mass is kept as was 1:3, the w/c ratio was fixed at 0.65, and the sand-to-binder ratio was kept 2.50. The material was mixed as per (IS 1727-1967), then cubes of 50 mm in size is casted. After casting, the cubes were stored at 23 ± 2 ˚C for 48 ± 2 h, Subsequently, the cubes were then demolded and placed in relative Humidity of 100% at 50 ± 0.5 ˚C until 8 days. Before testing, the cubes were allowed to cool to

23 ± 2 ◦C for 2 ± 0.25 h. A loading rate of 150 N/s was applied for testing of cubes.

RESULTS AND DISCUSSION

Properties of Weight Reduction and Particle Size of CPS by Calcination Conditions

Table 5 shows Max weight reduction and particle size of CPSs according to calcination Temperature. According to Table 5 weight of CPSs was reduced from 61.7% to 70.2% with increasing temperature and duration of calcination due to combustion of organic fibers, gases and water, etc. in raw sludge. Average particle size of CPSs was in the range of 17.87–39.56 μm, with decreasing trend with increasing calcination temperature up to certain temperature. as shown in Figure 7.

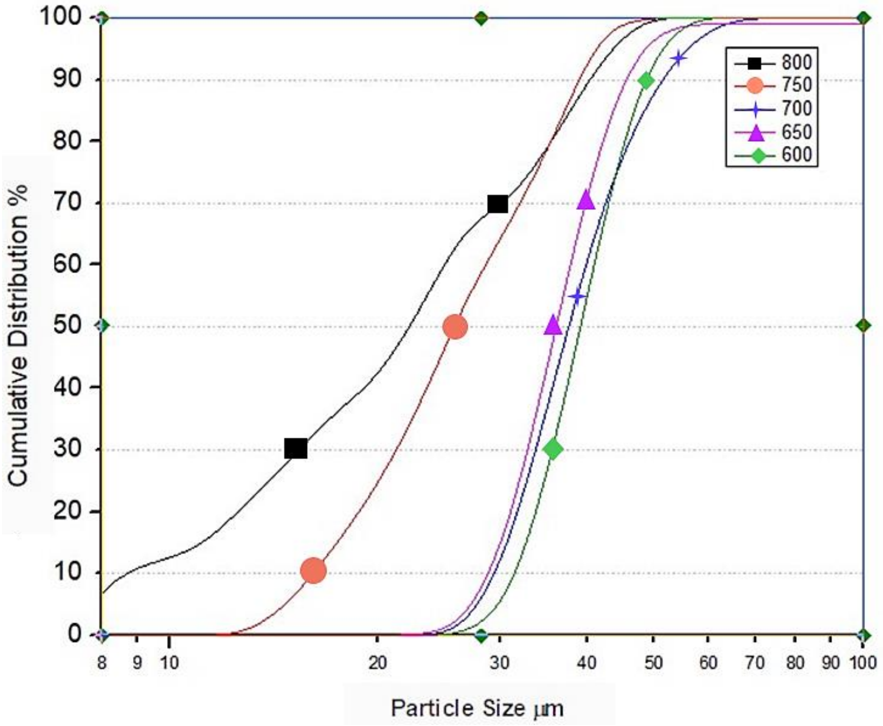


Figure 7. Particle size distribution of lime sludge at different calcination temperature.

Indirect Method

Two tests were performed for qualitative analysis of reactivity XRD and TGA.

Result of TGA-DTA

To determine the temperature sensitivity of various compound in Sludge and their qualitative quantification TGA-DTA test were performed on different samples Figure 8. The maximum mass loss is observed in Raw PS. The phase change occurs at around 750°C following the equation 2, after that weight loss is minimal. So, the maximum transformation occurs at 750°C. In this process calcite ($CaCO_3$) is converted in to Calcium oxide (CaO) and CO_2.

$$CaCO_3 \longrightarrow CaO + CO_2 \qquad (2)$$

Table 5 shows the results of the total weight reduction of paper sludge calcined at different temperatures. Raw Lime sludge contains moisture so there is a weight loss is seen. Conversion of Cao and CO_2 occurs at a temperature range of 700 to 800°C. phase transformation of the substance is observed. There is a decrease in amorphous content and an increase in recrystallized content observed after 800°C temp. The same results is also observed by the researcher [20].

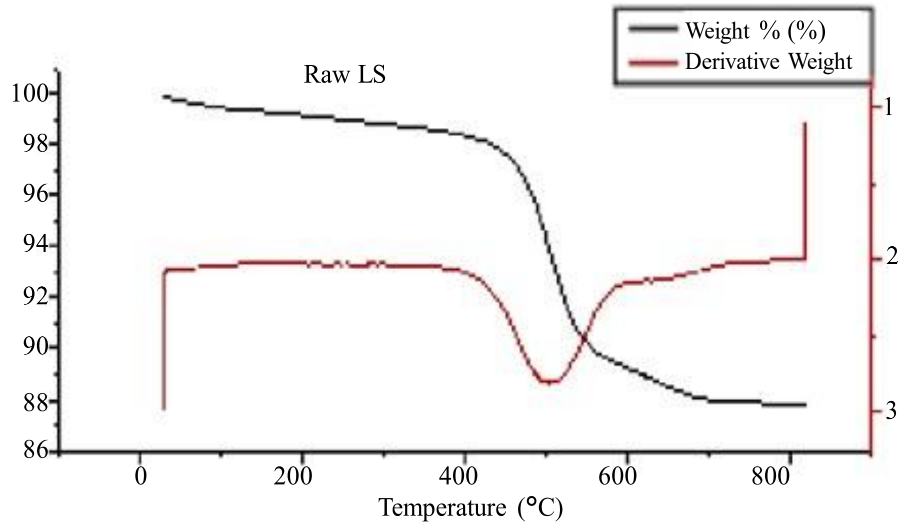
With an increase in calcination temperature, a reduction in particle size is also observed. Decrease In particle size also helps in increasing reactivity of the Sludge

Results of XRD

The Results of XRD is shown in Figure 9. The graphs were analyzed by MATCH 3 software to assess the composition and crystalline structure of the CPS. The mineral fraction of the dry Sludge is mainly calcite (65%) at angle (2ɵ) 31°, 42°, 48°, and kaolinite (7%) at angle 18°; other minerals are also present in small amounts, such as phyllosilicates (2%), 67°, Talk (4%) , and Quartz (3%). By Match 3 software analysis, it is found that as the temperature of calcinations increases the degree of crystalline decreases. The maximum decline occurs of about 24% for the samples calcined at 750°C for 2 hours. The indication of additional XRD peaks confirmed the phase transitions Higher temperature incineration of the Sludge promotes the recrystallisation of amorphous silica and decreases both the pozzolanic activity and the adsorption capacity of the resulting ash [27, 28, 29]. At greater calcination temperatures and lower angles, some CH was also found. Lime Sludge Calcined at optimum temp can be substituted by binder material due to the presence of CH, with additional source of silicate and aluminate such as FlyAsh and Metakaolin.

Table 5. Particle size and weight reduction of lime sludge at different calcination temperature.

Material	Temp	Max Weigth reduction (%)	Particle Size (µm)
Lime Sludge	Raw Lime Sludge	57.2	38
	600	61.7	34.5
	650	63.5	32.5
	700	65.3	28.9
	750	68.7	23.7
	800	68.9	19.6
0pc			17.5



(a)

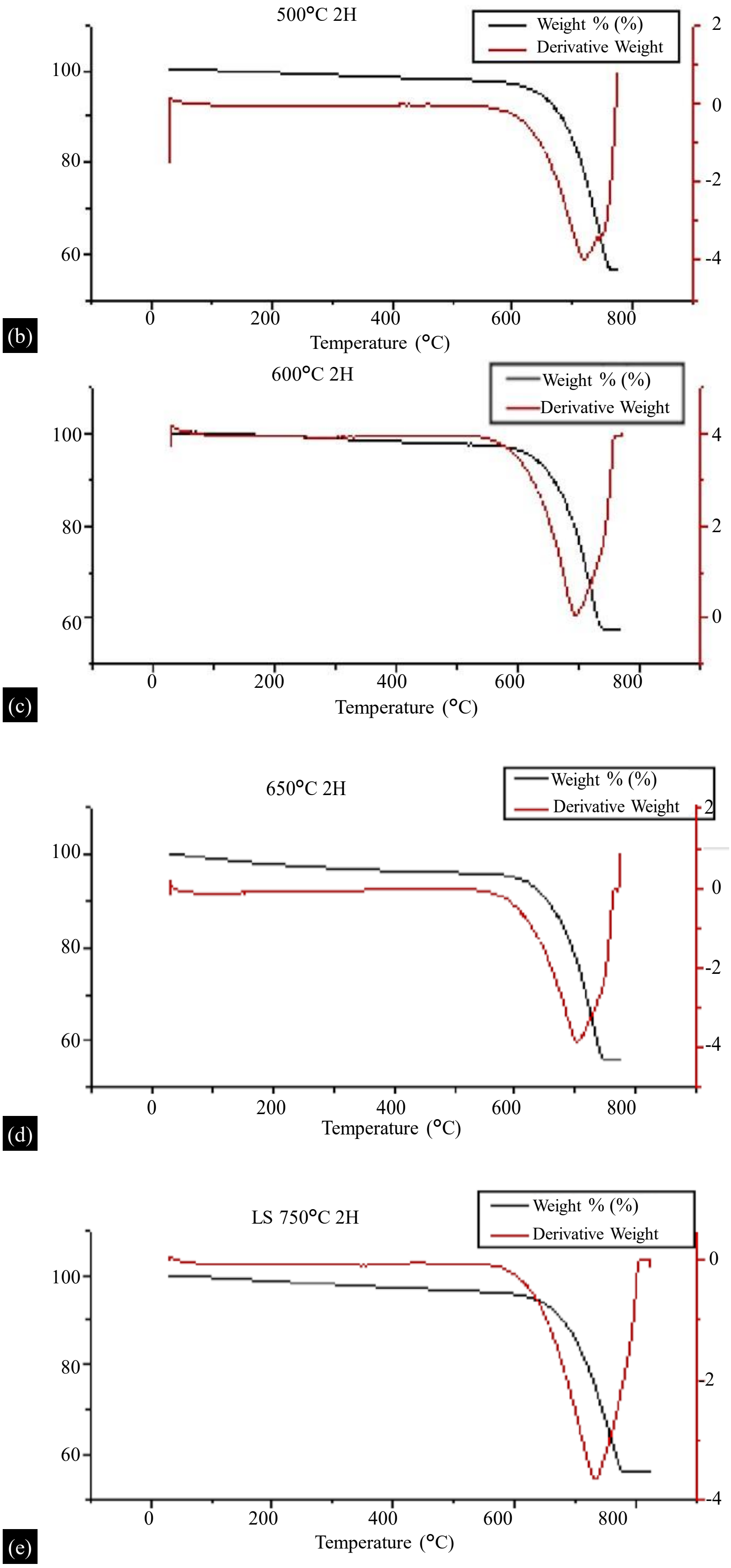


Figure 8. (a-e) TGA curve of lime sludge for different temp calcination.

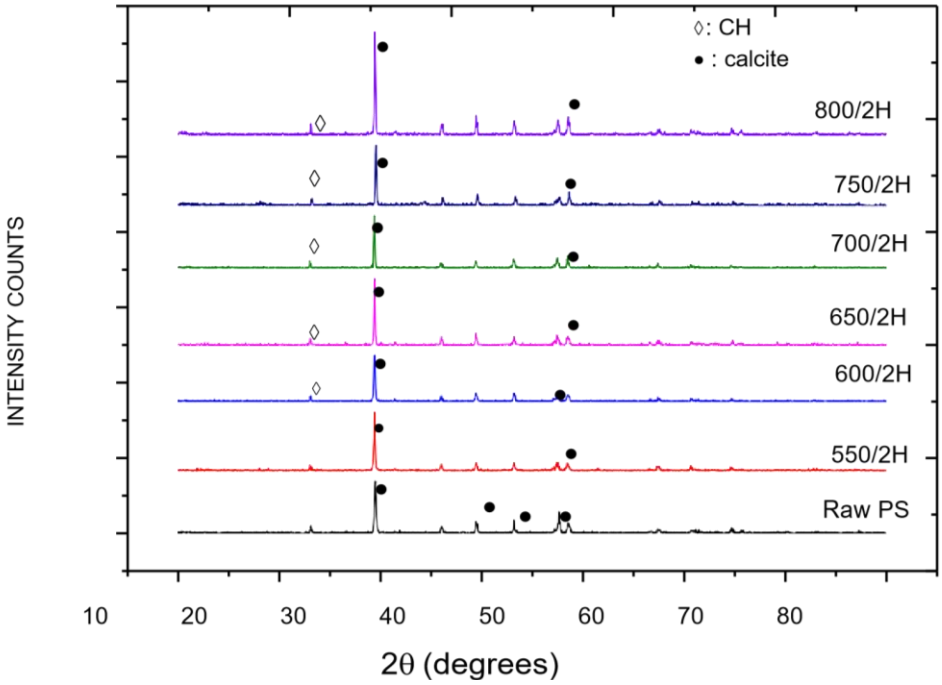


Figure 9. XRD pattern for raw PS, CPS at temperature range (550°C to 750°C) at lab scale.

Direct Method

To optimize the reactivity of CPS and Calcined clay percentage in the blend, Direct methods of determination of reactivity are adopted. Two tests were performed to optimize the temperature of calcinations and blend proportions.

Lime Reactivity Test

Lime Reactivity Test were performed according to (IS 1727-1967). The following Proportion was adopted for Lime Reactivity Test [26]. Here standard lime is replaced with Calcined paper sludge. The test is performed with CPS having different calcinations temperature. The W/b ratio is adjusted to get the desired flow.

Proportion-1:3M:9 (CPS: Calcined Clay: Sand)

M= Sp. Gravity of Calcined Clay/ Sp. Gravity of CPS

2.65/2.1= 1.3 Fixed

The Proportion used in the test is demonstrated in the Table 3

Sludge calcined at temperatures 600 and 650°C fails to give the desired result due to insufficient carbonation of $CaCO^3$. Results verified that the optimum temperature range for proper calcination of paper sludge is 750°C at 2h , as quoted by other authors [20, 30, 31] also. At 800°C and more due to, recrystallization of Sludge occurs. Crystallization affects the pozzolanic activity and thus demonstrates low strength.

Strength Activity Index Test

In this experiment, 30% of cement is replaced by calcined clay and calcined paper sludge binder with different proportions. 1:1, 1:2, 2:1 for mix M1, M2, and M3, respectively. Based on the previous studies [33] Lime reactivity test, the sludge is calcined at a calcination temperature of 750°C/2h and slow cooling conditions. Proportion and Results of SAI is tabulated in Table 4. M3 and M4 show good results in terms of replacement level. Non-availability of aluminosilicate ions to react with Ca+ ion is the possible reason for the low result of M2 [32, 33]

Table 6. Proportion used for lime reactivity test and results

S.N.	Activation Temperature	CPS	Calcined Clay	Sand	*Strength at 7 days (MPA)*
1	600	150	390	1350	5.2
2	650	150	390	1350	6.5
3	700	150	390	1350	7.31
4	750	150	390	1350	8.34
5	800	150	390	1350	8.03

Table 7. Proportion used for SAI test and results.

S.N.	Mix	Cement	CPS (750^0/2H)	Calcined Clay	Sand	W/b	7 day strength in Mpa	28 days strength Mpa	SAI % 7 days	SAI % 28 days
1	M1	100			300	0.5	21	36	100	100
2	M2	70	15	15	300	0.5	13.65	21.36	65	61
3	M3	70	10	20	300	0.5	14.70	29.52	70	82
4	M4	70	20	10	300	0.6	17.12	27.12	80.12	75

CONCLUSION AND DISCUSSION

The following Conclusions can be drawn from the present work:

1. The phase transformation of paper sludge affected by Calcinations temperature and thus affects the pozzolanic activity sludge.
2. The maximum weight loss for raw paper mill sludge occurs at 750°C after that weight loss becomes stable. Decrease in particle size with increase in calcination temp attribute to higher reactivity at high temperature.
3. The XRD analysis shows that paper mill sludge consists of mainly $CaCO_3$, Quartz, and Kaolinite. With an increase in temperature degree of crystallinity decreases and calcined sludge exhibit more reactivity. The maximum loss of crystallinity observed by the sample calcined at 750°C/2h of around 24%.
4. The above result is also validated by the Lime reactivity test, where Sludge calcined at 750^{o}C /2h shows better strength property at 7 days, table 6.
5. The SAI value suggested that CPS, Calcined Clay blend, exhibits pozzolanic properties and can become a promising binder to replace cement, Table 7.
6. Further studies are required to optimize the Proportion of replacement level of blend in cement.
7. The reactivity of CPS, Calcined Clay binder can be enhanced by using some precursor or alkali. Further studies needed to maximize the reactivity and optimize the replacement level so that waste sludge can be used more productively and its ill effect on the environment can be minimized

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