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The Effects of Using Eco-friendly Materials for the Production of High Strength Mortar

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A B S T R A C T

This study aims to evaluate the effect of using eco-friendly mineral admixtures, such as calcined clay (CC), silica fume (SF), and limestone (L), as partial replacements for ordinary Portland cement (OPC) in the production of high-strength mortar(HSM). The use of these materials can help reduce the environmental impact of cement production by decreasing carbon dioxide emissions and preserving natural resources. To achieve the desired strength, different mixtures were proportioned by increasing the percentage of binder and limiting the water-to-binder ratio (w/b) with the aid of superplasticizer (SP) type G. The mechanical behavior and strength of the blended cements were evaluated, and the optimal mix was determined based on the results of mechanical behavior, strength, and flowability. The results revealed that the optimal mix that gives the best mechanical behavior is the mix F32 with combination of (8% SF + 4 %L+ 13% CC) with an increase about 35 % to control mix strength. The study concludes that using CC, SF, and L as partial replacements for OPC can improve the properties of modern concrete/mortar mixes, resulting in improved durability, service-life properties, and mechanical properties. The results of this study can be utilized as a base for future studies on the same concrete mix design.

Introduction

Eco-friendly construction materials garner substantial interest for cost reduction and environmental preservation amid rising pollution [1],[2]. The clinker production process releases CO_2 emissions into the atmosphere [3]. Mass cement production exacerbates CO2 emissions, contributing to global warming and environmental problems. To combat this issue, mineral additives like limestone (L), calcined clay (CC), and silica fume (SF) are suggested as substitutes [4],[9],[10], enhancing the properties of modern concrete/mortar mixes by densification the microstructure and reducing Ca(OH)2 concentration through the pozzolanic reaction. This microstructure improvement boosts durability, service-life attributes, and mechanical properties. Calcined clays (CC) demonstrate effective pozzolanic activity when heated at temperatures between 500 and 800 °C [5],[6]. Silica fume (SF) acts as a filler in concrete, filling voids between cement particles similar to fine aggregates filling voids between coarse aggregates [7]. Plus, it contributes to the pozzolanic reaction. The chemical effect of SF in concrete is demonstrated by the reaction between amorphous silicon in SF and Ca(OH)2 in OPC, forming an additional binding material called calcium silicate hydrate (C-S-H), which is similar to the C-S-H obtained from hydrated OPC. SF is an effective supplementary cementitious material that enhances concrete's mechanical characteristics and durability [8],[9]. To use locally cheap materials, many studies have proved a good reaction between the alumina that the calcined clays have and limestone [10],[11] and its role in voids and pores filling.

The methodology can be summarized that to obtain high strength mortar, mixtures are to be proportioned by increasing the percentage of binder and limiting w/b by using SCMs and superplasticizer [12]. Superplasticisers are chemical admixtures added to concrete or/and mortar to improve its workability/ or flowability and reduce water content without affecting strength. Superplasticizers work by dispersing cement particles and reducing the viscosity of the concrete mix, allowing for better flow and compaction. This leads to a denser, stronger, and more durable concrete with improved durability and reduced permeability [13].

This study aimed to evaluate the influence of (OPC) replacement with CC, SF, and L, the eco-friendly SCM materials with the aid of SP, on the mechanical behavior of blended cement and

prepare a high-strength mortar from those materials.

Research Gap

Cement manufacturing is a process that requires significant energy and contributes to greenhouse gas emissions. Most cement factories in Iraq suffer from producing cement in traditional ways and neglecting the utilization of environmentally friendly materials during the production process. Researchers have explored using eco-friendly materials like fly ash, silica fume, and waste glass as cement additives to minimise this environmental impact. However, despite their potential benefits, a research gap exists regarding using locally available eco-friendly materials as additives. While industrial by-products have been studied as additives, they are not always available in all regions and can be expensive to transport. Therefore, it is necessary to investigate the possibility of using locally available eco-friendly materials, such as calcined clay and limestone, as additives in cement manufacturing. Further *exported* material is proposed in this study. It is a silica fume that is added in small percentages and enhances the properties of the cement blend.

Problem statement

1. In recent years, the global problem that has occupied the minds of many researchers is the problem of increasing carbon dioxide emissions and its current and future ramifications.
2. A site investigation was conducted on the Badoosh Cement factory plant/ Mosul. However, geological survey experts have shown a high proportion of high-carbonate materials(Limestone having $CaCO_3$ more than 80%) in an open-pit mine in Badoosh. These materials *alone are unsuitable* for cement production and must be mixed with other raw materials from the same quarry, which are present in very low proportions. This could render the *quarry unsuitable* for use in the distant future.
3. The current need is high-strength concrete incorporating these materials and modern superplasticizer.

Objective of the study

1. Determine whether these three materials (limestone(L) and calcined clay, which are locally available from the Badoosh quarry, and the exported material silica fume(SF)) meet the specifications.
2. Investigate whether these materials and SP are suitable to combine within several trial mixes by proposing alternate percentages of partial replacement by cement by suggesting two totals, 25% and 30%.
3. Determine the optimal mixes with the best mechanical behaviour, strength, and flowability results and whether those mixes have high-strength mortar characteristics.

4. Determine whether this best result may be achieved using *mostly available materials (CC+L) or vice versa.*
5. These results in mortar can be utilized as a base in future studies on the same *concrete* mix design.

Materials

Cement

Ordinary Portland cement (OPC) grade 32.5 R as per EN-197-1:2011[14]was used. It was sourced from the Badoosh extension factory and tested per Iraqi standard IQS:5/1984[15]. The chemical, physical, and mechanical properties are listed in Table 1.

Table 1. The physical and chemical tests of cement

Chemical analyses	IQS:5/1984 limits	Tested
CaO %		62.45
SiO_2		21.02
AL_2O_3		
Fe_2O_3		
SO_3		
MgO		
Insoluble residue%	≥ 1.5%	
Loss of ignition LOI	≥ 4 %	
Total		99.94
Free CaO %		1.43 %
L. S. F.		89.36
Main compounds (per bogue)		**Tested**
C3S		36.34
C2S		32.85
C3A		11.9
C4AF		8.0
Physical tests	**IQS:1984**	**Tested**
Fineness, blain method (cm^2/gm)	≥ 2250	2900
Setting time, inittial(min.)	≥ 45	120
Final (hrs.)	≤10	2:40
Soundness, autoclave	≤ 0.85	0.13 %
Water for consistency(g)		104
Compressive strength (Mpa)	**IQS**	**Tested**
@ 3 days	≥ 15	19.8
@ 7 days	≥ 23	24.5

Sand

River sand with a maximum aggregate size of (2.36) mm, supplied from the Kanhash area (east of Mosul, Iraq), was used in the study as fine aggregate. The specific gravity was determined according to ASTM C 128 [16], 2.62. The unit weight was 1670 kg/m^3, absorption was 1.4%, and the fineness modulus was 3.69. The grading of the sand satisfies the limitations mentioned in ASTM C33 [17]. The sand used in all study batches had been washed several times as per the ASTM C 117-95 [18] recommended. Also, the sand used was in the oven dry condition for all the mixtures, but SSD in all calculations. The gradation of sand is obvious in Table 2.

Table 2. Grading of sand

Sieve No.(mm)	Passing (%)	ASTM C33 Limits
No.4 (4.75)	100	95-100
No.8 (2.36)	90.7	80-100
No.16 (1.18)	64.1	50-85
No.30 (0.6)	43.2	25-60
No.50 (0.3)	20.7	5-30
No.100 (0.15)	7.7	1-10

Interground limestone

Limestone was sourced from the Badoosh quarry. It was subsequently milled to a particle size similar to, or smaller than, that of cement. The strength activity index was determined using ASTM C618 [19] and found 80%, and water demand 105%. The chemical characteristics of the limestone are presented in Table 3.

Table 3. The chemical compositions of limestone

Oxides	Content % (by mass)
$CaCO_3$	84
SiO_2	6.88
Al_2O_3	1.69
Fe_2O_3	0.98
MgO	2.53
Others	3.92

Calcined clay (CC)

The raw clay was obtained from the cement plant quarry in Badoosh, with a particle size ranging from 1 to 5 mm. It was initially ground and then calcined in a fixed furnace at a temperature of 800 ºC for one hour (based on recent studies [5], and due to its high carbonate content, it requires a higher temperature). It was then extracted, cooled, and ground to an appropriate fineness. Table 4. and Table 5. shows the physical properties and the chemical composition. CC satisfies the requirements of natural pozzolan type N mentioned in the standards of ASTM C618[19]. The strength activity index had been carried out according to the ASTM C618[19]. Figure 1. shows images of the calcined clay used, the furnace used for calcination, and other materials used.

Figure 1. Images of the furnace, calcined clay with sand.

Table 4. Chemical tests of CC.

Oxides	Content % by mass	Pozzolan class N ASTM C618
CaO	23.8	------
AL_2O_3	32.36	------
SiO_2	28.08	------
Fe_2O_3	4.53	------
SO_3	0.2	≤ 4%
2.53		------
Alkalies and others	3.6	
L.O.I	5.93	≤ 10%
$SiO_2+Al_2O_3+Fe_2O_3+CaO$	84.24	≥ 70 %

Table 5. Physical properties of CC.

Physical properties	Tested CC	ASTM C 618
Pozzolanic activity @ 28 days.	96 %	≥ 75 %
Water requirement w.r.t. control mix.	111 %	≤ 115 %
Blain fineness(cm^2/g)	4450	
Specific gravity	2.42	
Color	Reddish brown	

Silica fume(SF)

The activity index for strength was carried out using the ASTM C 1240 [20] guidelines, outlined in Table 6. Additionally, Table 7 displays the other properties of the SF, as reported by the manufacturer.

Table 6. . Pozzolanic Strength Activity Index Tests for SF.

Pozzolanic Strength Activity Index	Tested	Pozzolan class N ASTM C618
SAI at 7 days,	115%	≥ 105%
SAI at 28 days	105 %	
Water requirement with control	115	≤ 115 %

TABLE 7. Main properties of SF as per manufacturer.

Property	Value
State	Amorphous
Specific Gravity	2.3
Bulk Density (kg/m^3)	700
Chloride content	< 0.1%
Appearance	Grey Powder
Compatibility	Compatible with all types of cement, all SCMs, and SP.
Normal Dosage	5-8% up to 10%

Water

Ordinary drinking water in the lab has been used for mixing and normal curing processes. The water had no color or smell.

Superplasticizer (SP)

Sika® (ViscoCrete®-180G) was used. It was type G as per ASTM C 494[21]. It is light brownish in appearance and has a specific gravity of 1.065 ± (0.02) g/cm3. Its pH value ranges from 4-6. When using this admixture, as per *the recommendations of the manufacturer*, " it is important to follow standard Rules of good concreting practice for production and placing, and to conduct laboratory trials before concreting on site, especially when using a new mix design or producing new concrete/mortar components. *The recommended dosage is 0.4% - 2%* by weight of total cementitious materials. It should be added to the gauging water or into the concrete mixer. It should *not be added to dry cement.*" The manufacturer's recommendations were carefully taken into consideration.

Experimental works

Methodology

To achieve a high-strength mortar, the mixtures were proportioned by increasing the percentage of binder and limiting the water-to-binder ratio (w/b) with a superplasticizer (SP). This not only resulted in a significant increase in compressive strength but also prevented adverse effects in both the fresh and hardened states of the mortar. The focus was on reducing the w/b ratio to the point where the mortar remained workable and flowable rather than becoming stiff. Flowability tests and strength tests were conducted to ensure the desired flowability. The mix proportion of 1:2.2/0.3 was chosen for all the mixes, with two groups using supplementary cementitious materials (SCMs) for partial replacement of cement by weight: the first group used 30% and the second used 25%, along with different alternate percentages of a unique SP type but different in dosage to reach a target flow of 110±5. Trial mixes were carried out to achieve that flow. The control mix had proportions of 1:2.2/0.45. To determine whether the SCMs (SF, L, and MK) were active replacements, strength activity index tests were conducted by ASTM C 618[19]. A flowchart of work is clear in Appendix.

Mix proportions

The mixing proportions involved two categories of mixes: those with 30% partial percentage replacements and those with partial percentage replacements. The 30% replacements consisted of five mixes plus the control mix, while the 25% replacements consisted of four mixes plus the control mix.

In the first category of mixes, concerning (SCMs) only, the first mix, F11, with 6% SF, 6% Lime, and 18% CC. The second, F21, with 6% SF, 8% L, and 16% CC; the third, F31 with 8% SF, 8% L, and 14% CC; the fourth F41 with 10% SF, 8% L, and 12% CC; and the fifth F51 with 10% SF, 10% L, and 10% CC The second part of the mixtures consisted of four mixes: the first F12 with 5% SF, 3% L, and 17% CC; the second F22 with 6% SF, 4% L, and 15% CC; the third F32 with 8% SF, 4% L, and 13% CC; and the fourth F42 with 9% SF, 4% L, and 12% CC. The mix proportions are listed in Table 8.

Table 8. Mix proportions in kg/m^3.

Mix	Cement	Sand	Water	SF	L	CC	SP
F_o	618	1360	285	0	0	0	0
F11	480	1510	196	41	41	124	10.29
F12	480	1510	196	41	55	110	10.633
F13	480	1510	196	55	55	96	10.633
F14	480	1510	196	69	55	82	10.29
F15	480	1510	195	69	68	69	11.319
F21	515	1510	197	34	21	116	8.918
F22	515	1510	197	41	27	103	9.124
F23	515	1510	196	55	27	89	9.604
F24	515	1510	196	62	27	82	9.604

Results and discussions

The compressive strength of 50 mm cubes was tested at ages 7, 28, and 56 by ASTM C109 [22]. The average results of three cubes were taken at each age, with curing water temperature maintained for samples at 23±2°C. Flexural strength was measured using (40*40*160) prisms cast and cured at 23±2°C to meet the requirements of ASTM C192 [23], and the average of three prisms per age per mix was taken. Direct tension strength was tested using dog bone briquette specimens measuring 76mm in length, 44mm in width, and 25mm in thickness at ages 28 and 56 days under ASTM C190-85[24], with an average of three tests recorded. Flowability tests were conducted before casting the specimens according to ASTM C1437 [25], with the average of four crossed diameters results recorded for calculating flow %. The flow table apparatus used satisfied the requirements outlined in ASTM C230[26]. Table 9. and Figures 2-3 show the results obtained for flowability and compressive strength.

Table9. Results for flowability and compressive strength.

Mix	Flow %	$\sigma_{c.7}$	$\sigma_{c.28}$	$\sigma_{c.56}$
F_O	110	28.7	43.2	50.5
F11	110	37.4	44.6	67.6
F21	110	38.8	45.4	64.2
F31	115	41.4	46.3	67.3
F41	118	41.2	45.0	63.8
F51	103	41.6	43.8	61.5
F12	112	37.7	43.4	63.4
F22	105	40.9	48.6	67.6
F32	110	43.4	50.0	68.3
F42	104	45.2	48.3	63.4

Where: σ_c.7,$\sigma_{c.}$28, σ_c56 are compressive strength results in (Mpa) corresponding to 7,28, and 56 days.

Table 10. Results for direct tension and flexure strength in (Mpa).

Mix	$\sigma_{t.}28$	$\sigma_{t.56}$	$\sigma_{f.}28$	$\sigma_{f.}56$
Fo	2.9	3.18	7.3	7.85
F11	3.16	4.52	8.23	11.24
F21	3.13	4.26	7.87	10.65
F31	3.26	4.38	8.07	11.26
F41	3.14	4.35	8.56	10.65
F51	3.06	4.31	8.42	10.32
F12	3.05	4.2	8.29	10.82
F22	3.42	4.16	8.3	11.21
F32	3.25	4.37	8.34	11.50
F42	3.19	4.42	8.45	11.62

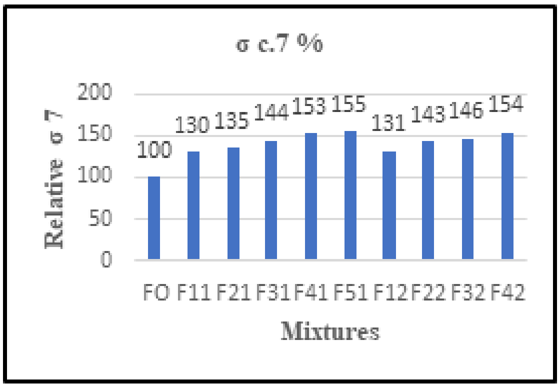

Figure 2. Relative compressive strengths at 7 days with Respect to control mix (Fo).

According to the results presented in Figure 2, the 7-day compressive strength of mortar increases with the addition of SF up to 9-10%, starting from 5-6% SF content, accompanied by a relative decrease in CC content in the mixes. This increase in compressive strength is due to the high reactivity of SF at *early* ages and its ability to strengthen the transition zone and bond between cement paste and aggregate.SF reacts with the hydration products of cement, Ca(OH)2, to form a C-S-H gel with a morphology that varies from crystalline fibers to a reticular network, with very small gel pores size and a very low surface area of 100 to 700 m2/g. This reaction not only results in a higher strength of the gel but also clears or relieves the mortar microstructure from the weak hexagonal-prism morphology of Ca(OH)2, one of the hydration products, leading to a more dense, low porous, and stronger mortar. The addition of pozzolans, such as SF, to mortar can also help in resisting sulfate attack by refining the pores[12],[27],[28].

Figures 3 and 4 present the compressive strength results at 7, 28, and 56 days for mixes F_O to F51 and F to F42, respectively.

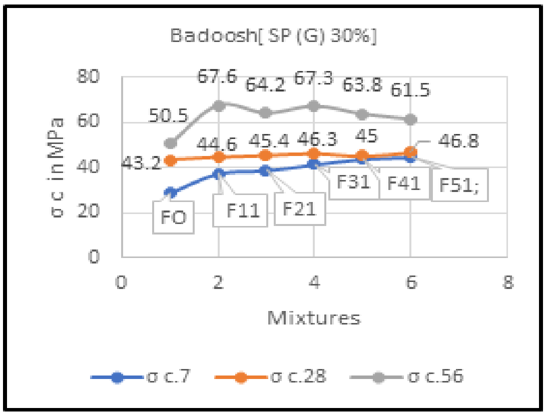

Figure 3. Compressive strength in (Mpa) for mixes Fo, F11 to F51 total 30% replacement.

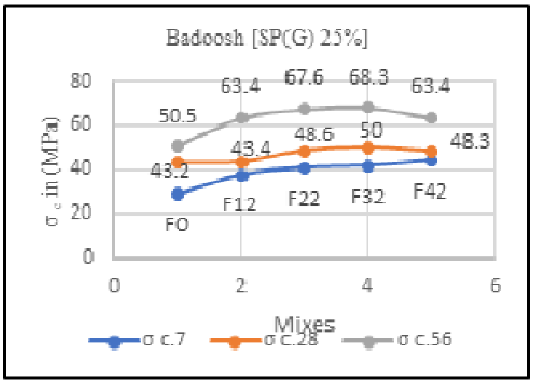

Figure 4. Compressive results (Mpa) at 7,28, and 56 days, total 25% replacement.

It is evident from Figures 3 and 4 that there is a slight development in values at 28 days of age, which may be attributed to the relatively low fineness of the calcined clay & and limestone, which negatively affects the pozzolanic reaction rate. As shown in the figures, the behavior became clearer at 56 days of age, and the values' differences became more pronounced. For the first two mixes, F11 and F12, it was observed that the relative increase in strength in mix F11 was higher. Mix F11 contained 6% limestone, which is believed to have saturated the CC oxides(especially aluminate) more than the corresponding mix with a 3% limestone content[29,30,31], in addition to the pozzolanic reaction. Increasing the limestone (L) content from 3% to 4% and reducing the calcined clay (CC) content from 17% to 15% and then to 13% in mixes F22 and F32 increased in strength, with the optimal value observed for mix F32. The L/CC ratio in mix F32 was close to that of mix F11, which had a 1:3 ratio. This supports the author's view that the L/CC ratio equal to 1:3 is optimal for the reaction between limestone carbonates and the alumina phases in the binder material, including the calcined clay, which produces carbo aluminates that contributes to strength development by filling the voids[10]. The latter justification may be the reason for the decrease in strength observed in F21 compared to F11, where the binder content was reduced by 4%, whereby L increased by 2% above the optimal ratio and CC decreased by 2% below the *expected* optimal ratio. In other words, the increase in the optimal level of limestone for a *reduced* amount of alumina in CC resulted in two unreacted components, which have less strength contribution, only fulfilling the role as fillers [32] replacing C3S and C2S contents, which were lost due to an inappropriate replacement in this trial mix.F21. As for Figure 3, it can be observed that mix F32, the third trial mix, resulted in an increase in strength compared to F22. This may be attributed to the effective filling of pores by SF. However, the strength value decreased in mix F51, possibly due to the excess unreactive ratio of SF, L, and CC.

Regarding flowability tests, the results have cleared that the significant factor affecting the results was the percentage of SF used. When SF was used as a replacement at 10%, the water demand increased to 115%. Similarly, when CC was replaced at 20%, the water demand increased to 111%. On the other hand, when limestone was used as a replacement at 20%, the water demand increased to 105%. In any case, none of these combinations can be used in mixtures without adding SP. The test results have shown that all the mixtures failed in the *flowability* test and appeared stiff. Figure 5 illustrates the flow of one of the mixtures without using SP.

Figure 5. An image of the flow of the mixture without SP.

Regarding the tension test results, both the direct and flexure tensile tests showed a proportional relationship with the corresponding compressive strength. However, there was no direct proportionality. These results were consistent with what P. Kumar explained in his book [27] pp.76. This was due to the heterogeneous structure of concrete, which gains and maintains its strength through bond stress created by cement or cementitious materials due to the hydration reactions between the cement matrix, hydration products, and transition zone. The difference among compressive, tensile, and flexure strengths is only due to the failure mechanism, which varies depending on the loading condition in each case. Flexure involves only a small part of the specimen near the bottom of the specimen being subjected to high stresses, and the forces generated are a mix of tension, compression, and shear. Direct tensile involves the entire volume of the specimen being subjected to applied pure tension, and compressive strength involves pure compression. Concrete/mortar is weak in tension and strong in compression due to the axiomatic fact. *Furthermore*, the results showed that as the compressive strength of any mix in the study increased, both tensile and flexural strength increased but at a decreasing rate. Also, as the age increased, the rate of increase became less. However, all the results indicated higher values than the control mixtures due to the enhancement of the microstructure of mortar mixtures by the admixture SP, a decrease in w/b, and the addition of SCMs.

Conclusions

1. In the laboratory, HSM can be prepared using mostly locally available materials such as clay and limestone in the Allan quarry/ Badoosh.
2. At the industrial level, calcined clay can be manufactured by calcinating that local clay through a dedicated production line, which produces less CO_2 emissions and at a lower cost, and then added to a cement mill by, as per the study, 18% of clinker mass. At the same time, limestone can be *added only* 6% of clinker mass to the cement mill during clinker grinding. Surely, this means less environmental pollution and lower cost.
3. Using 6% SF + 6% L + 18% CC for 30% partial replacement, which is *mostly available in cheap, eco-friendly materials,* and 8% SF + 4% L + 13% CC for 25% partial replacement by cement weight give the highest level of strength. There is a relative decrease in strength in other HSMs at 25% or 30% of total levels prepared differently.
4. The maximum increase in compressive strength compared to the control mix is about 55% on the seventh day and a 35% increase on the 56th day.
5. With increasing compressive strength, tensile strength also increases. However, there is no direct proportionality between them. Also, the increase in tensile strength occurs slower than compressive strength.
6. Using the appropriate new version of the superplasticizer material, the polyhydroxy carboxylate base significantly improves HSM's fresh properties. SP reduces water demand by more than 55%. SP significantly improves the flowability of HSM.
7. The effectiveness of SP begins after 13-14 minutes of continuous mixing and continues for another 13-15 minutes; then, the flow loss, or so-called slump loss, occurs.
8. Using these mixes without SP is not recommended as water is significantly absorbed by SCMs, and the mixes become non-flowable.

Recommendations

1. The science of building materials engineering is concerned with studying and examining building materials, their physical, chemical, and mechanical tests... and the fascinating and abundantly available local clay. Our laboratory capabilities in burning clay are limited to burning and calcining it in fixed furnaces. Cement manufacturing is another science that focuses on the production process. It is an advanced science with its sophisticated tools and controlled rotary furnaces, with controlled temperature and oxygen levels, and periodic examination of the degree of calcination by their laboratories, which should ideally be 100% or 99% calcination and not exceed those ratios so as not to have a negative impact.

2. According to the site investigation, developing an old laboratory may be difficult. Still, it is possible and recommended to establish an integrated production line to produce calcined clay, starting with the crusher, then the mill, then the rotary furnace, and ending with special storage silos. On the other hand, feeders are also necessary for the building design of the cement mills to, include clinker, gypsum, calcined clay, limestone, and silica feeders.

Joint *coordination and cooperation* between the parties are essential to achieve more satisfying results.

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Appendix: Flowchart of the work.