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Journal Of Surveying And Structural Engineering

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Contents

Use Of Copper ORE Tailings - As An Excellent Pozzolana In The Preparation Of Concrete

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ABSTRACT

Industrialization extremely demands to the uplift of nation's economy. However, it causes severe environmental pollution due to the generated waste materials. Copper ore tailings, is a waste material obtained in the copper mines, after the extraction of copper concentrate from the ore. Some trace percentage of copper will be left in the ore after the extraction process is completed. This copper ore tailing is produced in large quantity in the copper mines. Disposing of this copper ore tailing from the mines premises is a big head ache for the concerned authorities. In this experimental work an attempt has been made to study the suitability of copper ore tailings as an admixture in the preparation of concrete by replacing the cement in different percentages viz., 0%, 10%, 20%, 30%, 40% and 50%. Compressive strength and Water absorption test were conducted on the prepared specimens. The results show that the replacements of ordinary Portland cement by copper ore tailings safe up to 20% considering average minimum field strength. If characteristic strength is considered replacement copper ore tailings upto 30% may be considered as safe and the water absorption decreases at 20% copper ore tailings content and increases for all other copper ore tailings content.

Key words: Copper tailings, Compressive strength, Water absorption

1.0. INTRODUCTION

Due to the development and increment in the construction industry, the scarcity of the natural resources and building materials has been on a very large scale and the demand for the natural resources and materials is increasing day by day. At the same time, disposal of industrial waste or by-products has become more difficult and expensive as a result of the increasing stringent environmental regulations and shortages of suitable, nearby disposal sites. Industrial by-products also creates environmental hazard as they may be toxic for environment. So the usage of industrial by-products and solid waste materials as aggregates in concrete and in the soil stabilization for road and other type of the construction

work is the need of the hour. Some of the mines wastes at the various places throughout the country are copper ore tailings, iron ore tailings, manganese ore tailings, gold ore tailings, zinc ore tailings, lead ore tailings, metal extracts etc[1, 2, 5].

Copper ore tailing is the fine gray type sandy material and it has some acidic smell. It can damage the root of the plants by mixing heavy metal contaminants in the soil. Copper ore tailing wastes, even if treated, contain heavy metals with hazardous properties posing environmental risks for disposal. The heavy metals present in copper tailing may leach into ground water resulting in contamination. Copper ore tailing utilization, especially in concrete, has significant environmental benefits including, increasing the life of pavements and structures by improving stability of soil, reduction in the adverse air emissions when used in copper tailing bricks and stabilized blocks, etc. According to the chemical classification report, copper ore tailing is a good quality class-N natural pozzolana. So we can use copper tailing as a pozzolanic material in the construction industry and in suitable places. Copper ore tailing is available free of cost and only transportation expenses are there [1, 2, 5].

Copper ore tailing is one of the waste by-products, produced by the Ingaldal Copper Mines, a unit of the Hutti Gold Mines Company Ltd., situated at a distance of 10 Kms from Chitradurga, Karnataka. More than 1, 50, 00cu-m of copper ore tailings had been dumped in Ingaldhal. It has become a problem for concerned authorities (H G M limited, Ingaldhal), to dispose off the waste which is more alkaline in nature (pH is above 13), which is unfit for farming.

The main aim of this experimental work was to use of copper ore tailings as an admixture by replacing the cement in different percentages viz., 0%, 10%, 20%, 30%, 40% and 50%, in preparation of concrete.

2.0. EXPERIMENTAL WORK

2.1. MATERIALS USED

Cement: The cement used in the experimentation was ordinary Portland cement-43 grade, which satisfies the requirements of IS: 8112-1989 specifications. The physical properties of tested cement are given in Table No. 2.1.1

Coarse aggregates: The crushed stone aggregate were collected from the local quarry. The coarse aggregates used in the experimentation were 10mm and down size aggregate and tested as per IS: 383-1970 and 2386-1963 (I, II and III) specifications. The aggregates used were having fineness modulus

1.9. Sieve analyses of coarse aggregate are given in Table No. 2.1.2 and physical and mechanical properties of tested coarse aggregates are given in Table No.2.1.3

Fine aggregates: Locally available sand collected from the bed of river Bhadra was used as fine aggregate. The sand used was having fineness modulus 2.96 and confirmed to grading zone-III as per IS: 383-1970 specification. Sieve analyses of fine aggregate are given in Table No. 2.1.4 and physical properties of tested fine aggregate are given in Table No. 2.1.5

Copper ore tailings: Copper ore tailing used in the experimental program was procured from Ingaldal Copper Mines, a unit of the Hutti Gold Mines Company Ltd., under dry mode of condition. The chemical and physical properties of tested copper ore tailing are given in Table No. 2.1.6 and Table No. 2.1.7

Water: Ordinary potable water free from organic content, turbidity and salts was used for mixing and for curing throughout the experimental work.

Table 2.1.1: Physical properties ordinary Portland cement 43-grade (IS: 8112-1989)

Properties	Results	Permissible limit as per IS: 8112-1989
Fineness	28.1 m^2/N	Should not be more than 22.5 m^2/N
Normal consistency	29.7	-
Specific gravity	3.15	-
Setting time		
a. Initial	170 Min	Should not be less than 30 Minutes
b. Final	273 Min	Should not be more than 600 Minutes
Soundness test		
a. Le-chat expansion	1	10mm maximum 0.8% maximum
b. Auto clave%	0.09	
Compressive strength of mortar cubes for		
a.3days b.7days	24.5N/mm^2	Should not be less than 23 N/mm^2
c.28 days	36 N/mm^2	Should not be less than 33 N/mm2
	46.5N/mm^2	Should not be less than 43 N/mm2

Table 2.1.2: Sieve analysis of coarse aggregate (IS: 383-1970)

IS sieve size	Weight retained (grams)	Cumulative weight retained (grams)	Cumulative percentage weight retained	Cumulative percentage passing	ISI permissible limit
12.5mm	0	0	0	100	100
10mm	0	0	0	100	85-100
4.75mm	1860	1860	93	7	0-20
2.36mm	93	1953	97.65	2.35	0-5
pan	47	2000	-	-	-
Total	2000	-	190.65	-	-
Fineness modulus = 190.65/100 = 1.9					

Table 2.1.3: Physical and Mechanical properties of coarse aggregate (IS: 2386-1963)

Properties	Results	Permissible limit as per IS: 2386-1963
Impact value	15.50%	Should not be more than 30% used for concrete
Crushing value	25%	Should not be more than 30% for surface course and 45% other than wearing course
Specific gravity	2.65	In between range 2.6-2.8
Moisture content	0.16%	-

Table 2.1.4: Sieve analysis of fine aggregate (IS: 383-1970)

IS sieve size	Weight retained (grams)	Cumulative weight retained (grams)	Cumulative percentage weight retained	Cumulative percentage passing	Grading zone III
10	0	0	0	100	100
4.75	5	1	99	-	90-100
2.36	44	45	9	91	85-100
1.18	30	75	15	85	75-100
600ì m	50	125	25	75	60-79
300ì m	185	310	62	38	Dec-40
150ì m	120	430	86	14	0-10
Pan	70	500	-	-	-
Total	500 gm	-	296	-	-

Fineness Modulus: 296/ 100 = 2.96

Table 2.1.5: Physical properties of fine aggregate (IS: 2386-1963)

Properties	Results	Permissible limit as per IS: 2386-1963
Organic impurities	Colourless	Colour less /Straw Colour/Dark Colour
Silt content	0.70%	Should not be more than 6-10%
Specific gravity	2.63	Should be between the limit 2.6-2.7
Bulking of sand	16%	Should not be more than 40%
Moisture content	0.65%	-

Table 2.1.6: Chemical properties of copper ore tailings

Properties	Test results
Loss on ignition(L.O.I.)	2.19
Silica(SiO2)	71.52
Magnesium oxide(MgO)	0.49
Calcium oxide(CaO)	0.16
Aluminium oxide(Al2O3))	13.96
Iron oxide (Fe2O3)	3.64
Potassium oxide(K2O)	1.82
Sodium oxide(Na2O)	4.12
Titanium oxide(TiO2)	0.013
Copper oxide(CuO)	0.32
Manganese oxide (Mn2O2)	0.072
SiO2+AL2O3+Fe2O3	92.12

Table 2.1.7: Physical properties of copper ore tailings

Properties	Test results
Specific gravity	3.1
Fineness	6.70 to10.20 %
Standard Consistency	27.50%
Compressive Strength	zero
pH	13.6

2.2. EXPERIMENTAL PROCEDURE

The concrete ingredients namely cement, fine aggregate (sand) and coarse aggregate (jelly) were weighed according to their proportion and they were dry mixed on non-absorbent plat form and mixed thoroughly in dry state and required quantity of tailings was added in dry condition and mixed again thoroughly. To this, the calculated quantity of water was added mixed rigorously and homogeneously. Before any fresh concrete was poured into the concrete moulds, all concrete moulds were cleaned from the existing concrete stain and oil was applied inside the moulds.

The fresh concrete was placed into the mould with the help of scoop. The moulds were filled with concrete in workability condition in three layers each layer being compacted by using standard tamping rod thoroughly and vibrated using table vibrator to achieve an adequate compaction. After adequate compaction, the specimens were finished smooth and left. After 24 hours, the specimens were demoulded and transferred to curing tank where in they were allowed to cure for 28 days.

Concrete specimens for compressive strength test were of dimensions 150mm x 150mm x 150mm. The specimens were placed in between the platens of a compression-testing machine. Load was applied gradually until the specimen fails.

The test was conducted after the concrete specimens were cured for 28 days. The test procedure was carried out accordance with Indian standard: 516-1959 specification.

The compressive strength of concrete can be calculated using the following formula:

$fc = P/A$

Where, fc = Compressive strength of concrete.

P = Maximum load applied to the specimen.

A = Cross sectional area of the specimen.

3.0. EXPERIMENTAL RESULTS -

The following Table No. 3.0.1 and Table No. 3.0.2 give the details of the experimental result.

Table 3.0.1: Compressive strength test results of concrete with different percentage replacement of cement by copper tailings

Mix ratio	Percentage replacement of cement by copper tailings	Compressive strength in N/mm ^{2} for different curing period in days		
		7	14	28
	0	9.7	13.9	14.45
M10	10	9.2	13.1	13.7
1:03:06	20	8.5	12.2	12.8
W/C	30	6.9	9.9	10.6
0.6	40	6.3	8	8.7
	50	5.75	7.11	7.14
	0	16.11	21.04	22.63
M15	10	13.62	18.15	20.03
1:02:04	20	12.45	17.6	18.75
W/C	30	11.65	15.6	16.9
0.55	40	9.4	13.4	14.25
	50	7.58	11.51	12.01
	0	24.12	31.56	33.94
M20	10	20.43	27.22	30.14
01:05.5	20	18.67	26.4	28.12
W/C	30	17.47	23.4	25.35
0.5	40	14.1	20.41	21.37
	50	11.38	17.02	18.01
	0	36.18	47.34	50.91
M25	10	30.64	40.83	45.21
1:01:02	20	28	39.6	42.18
W/C	30	26.2	35.1	38.02
0.45	40	18.33	26.53	27.78
	50	12.82	20.05	21.3

Table 3.0.2: The water absorption test results of concrete with different percentage replacement of cement by copper tailings

Mix ratio	Percentage replacement of cement by copper tailings	Percentage water absorption for different curing Period in days		
		7	14	28
	0	2.34	3.07	2.92
M10	10	2.89	2.47	2.31
1:03:06	20	2.55	2.38	2.17
W/C	30	2.09	1.97	1.89
0.6	40	2.02	1.79	1.69
	50	1.93	1.62	1.51
	0	2.65	2.35	2.2
M15	10	2.55	2.23	2.08
1:02:04	20	1.99	1.97	1.81
W/C	30	1.96	1.64	1.53
0.55	40	1.72	1.54	1.19
	50	1.69	1.44	1.11
	0	2.75	2.65	2.4
M20	10	2.65	2.43	2.38
01:05.5	20	1.59	1.37	1.31
W/C	30	1.46	1.34	1.23
0.5	40	1.32	1.31	1.18
	50	1.22	1.19	1.08
M25	0	2.76	2.46	2.4
1:01:02	10	2.65	2.33	2.37
W/C	20	1.69	1.39	1.41
0.45	30	1.56	1.34	1.39
	40	1.52	1.31	1.29
	50	1.12	1.11	1.07

4.0. OBSERVATIONS AND DISCUSSIONS

Based on the experimental results the following observations were made

1. It has been observed that from the experimentation, strength of concrete blocks decreases with increase in the percentage of tailings. The cement can be replaced by copper tailings upto 30%.

2. It has been observed that the addition of copper tailings as replace to ordinary Portland cement causes reduction in the compressive strength of concrete.

3. It has been observed that the replacement of ordinary Portland cement by copper tailings safe upto 20% considering average minimum field strength. If characteristic strength is considered replacement upto 30% may be considered as safe.

4. It has been observed that the increase in replacement of ordinary Portland cement cause decrease in water absorption and consequently enhancing the durability of the concrete, which can be considered as a favourable phenomenon

5.0. CONCLUSIONS

The following conclusions can be drawn from the results obtained from the experimental work carried out.

1. It can be concluded that the feasibility of utilization of copper tailings improves the properties of concrete has been identified in this experimental work.

2. It can be concluded that the water absorption decreases at 20% copper tailings content and increases for all other copper tailings content.

3. It can be concluded that the replacement of ordinary Portland cement by copper tailings proves to be economical by 17.28%.

4. Utilization of copper tailings for partial replacement of cement on concrete solves the problem of dumping down the waste materials

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a. IS: 3812-1981 Specification for fly ash for use as pozzolance and admixture.
b. IS: 6461 (part-II)-1972 Glossary of terms relating to cement concrete.
c. IS: 6491-1972 Method of sampling fly ash.

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Use Of Copper Slag In Concrete

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ABSTRACT

The present investigation assesses the incorporation of copper slag in concrete. The effect of copper slag as partial replacement of cement on the compressive strength of concrete has been investigated. The hydration of cement with copper slag was investigated through X-ray diffraction (XRD). Five concrete mixes (C0, C5, C10, C15 and C20) were made by replacing cement with 5%, 10%, 15% and 20% of copper slag by mass respectively. The water/cement ratio in all the mixes was kept at 0.43. Results showed that the compressive strength of concrete decreases as CS content increases for all curing ages. The reduction in compressive strength is minor up to 10% of CS but beyond 10% of CS, there is significant reduction in compressive strength due to the increase in free water content in mixes. XRD showed that the 10% of copper slag slightly reduces the hydration of cement, but 20% of copper slag significantly reduces the hydration of cement. It indicates that the copper slag can be utilized as supplementary cement replacement material in concrete. The optimum content of copper slag as replacement of cement is recommended as 10%.

Keywords: Alite, Compressive strength, Concrete, Copper slag, Hydration, Portlandite, Quartz, X-ray diffraction

1.0. INTRODUCTION

In view of global warming, efforts are on to reduce the emission of CO_2 to the environment. Cement industry is a major contributor in the emission of CO_2 as well as in using up high levels of energy resources in the production of cement. Researchers from all over the world are focusing on the ways of utilizing industrial waste, as supplementary cement replacement materials. This waste utilization would not only be economical, but may also help protecting the environment. Industrial wastes, such as slag, fly ash and silica fume are being used as supplementary cement replacement materials [1].

Copper slag is one of the material that is considered as a waste material which could have a promising future in construction industry as partial or full substitute of either cement or aggregates [2]. It is a by-product obtained during the smelting and refining of copper. During smelting, a molten pool of copper forms at the bottom of the furnace while a layer of impure metal form a less dense liquid floating on top of the copper melt, which is the slag. The molten slag is discharged from the furnace at 1000–1300°C. If the molten slag is water quenched, a glassy copper slag is obtained. To produce every ton of copper, approximately 2.2-3 tons copper slag is generated and approximately 24.6 million tons of slag is generated from the world copper industry [3]. Therefore, numerous contemporary researches have focused on the application of copper slag in cement and concrete production as a suitable path towards sustainable development. The use of copper slag in cement and concrete provides potential environmental as well as economic benefits for all related industries, particularly in areas where a considerable amount of copper slag is produced.

The present investigation encourages the utilization of industrial waste copper slag in concrete and studied its effect on the properties of concrete for obtaining a supplementary cement replacement material.

2. LITERATURE REVIEW

Many researchers have investigated the use of copper slag as partial replacement of ordinary Portland cement. The use of copper slag reduces the early age strength (1 day) while increasing it beyond 7 days [4]. The use of ground copper slag up to 15% by mass as a Portland cement replacement increased the strength significantly [5]. The addition of copper slag to cement increased its initial and final setting times [6]. The concrete batches with copper slag addition presented greater mechanical and durability performance [7].Blends of copper slag with Portland cement generally possess properties equivalent to Portland cement containing fly ash [8].

3. MATERIALS AND METHODS

3.1. MATERIALS USED

Ordinary Portland cement (OPC) of 43 grade was used. It was conforming to BIS 8112 [9]. The physical properties of OPC are given in Table 1. The fine aggregate used was locally available river sand having a 4.75 mm nominal maximum size. The sand was conforming to grading zone II as per BIS 383 [10]. The coarse aggregate used was crushed stone having a 20 mm nominal maximum size. The

properties of coarse aggregate and fine aggregate are given in Table 2. Fresh and clean tap water was used. The water was conforming to the requirements of water for concreting and curing as per BIS 456 [11]. Copper slag obtained from Synco Industries Limited (Jodhpur, Rajasthan) was used. The physical and chemical properties of copper slag are given in Table 3.

3.2. CONCRETE MIXES AND MIX PROPORTIONS

In this work, one control mix C0 was designed as per Indian Standard Specifications BIS 10262 [12] for M25 grade of concrete. The other four concrete mixes (C5, C10, C15 and C20) were made by replacing cement with 5%, 10%, 15% and 20% of copper slag by mass respectively. The water/cement (w/c) ratio in all the mixes was kept at 0.43. Mix proportions of concrete mixes are given in Table 4.

3.3. PREPARATION AND CASTING OF TEST SPECIMENS

Compressive strength of concrete is determined from cube specimens of 150 mm X 150 mm X 150 mm in size. All the specimens were prepared in accordance with Indian Standard Specifications BIS 516 [13]. After casting, test specimens were covered with plastic sheets and left in the casting room for 24 hours at a temperature of about 27±2oC. The specimens were removed from the moulds after 24 hours of casting and put into a water-curing room until the time of the test.

3.4. WORKABILITY OF CONCRETE

Workability of concrete was tested using compacting factor test apparatus as per BIS 1199 [14]. The workability of concrete was determined immediately after preparing fresh concrete.

3.5 Compressive strength of concrete

Concrete cube of size 150 mm X 150 mm X 150 was tested for compressive strength as per BIS 516 [13]. For the compressive strength test, a loading rate of 2.5kN/s was applied. The test was performed at 3, 7, 14 and 28 days of curing. Specimens were tested immediately on removal from the water and while they are still in wet condition, surface water and grit shall be wiped off the specimens and tested.

3.6. X- RAY DIFFRACTION TEST

The X- ray diffraction test was performed on samples of cementitious powder of mixes C0, C10, and C20. The samples of cementitious powder were collected from the remnant of concrete specimens after 28 days compressive strength test. The X-ray diffractograms of different samples were recorded on Panalytical X'Pert PRO with Bragg–Brentano geometry. Cu Kα radiation was used with a wavelength (λ= 1.54060 Å). X-ray tube was operated at 45 kV voltage and 40 mA current. Powder samples were loaded on aluminum sample holder having dimensions 2cm X 1·5cm X 0·2 cm. The measurements were carried out in a 2θ range of 10.0066° to 99.9846° with a step width of 0.0130°.

4. RESULTS AND DISCUSSION

4.1. WORKABILITY

The value of compacting factor for each mix is given in Table 4. The compacting factor of concrete with different replacement levels of copper slag is also shown in Figure 1. It can be observed that the workability of concrete increases as copper slag content increases as shown in Figure 1. The compacting factor for 0% copper slag was 0.849, while compacting factor for 20% copper slag was 0.925. The addition of copper slag increases the workability of concrete due to its glassy surface which reduces its water absorption. Due to the low water absorption of copper slag, free water content increases in the mix and hence, it is observed that workability of concrete has increased.

4.2. COMPRESSIVE STRENGTH

The compressive strength of concrete was determined at the curing ages of 3, 7, 14 and 28 days. Results are given in Table 5 and shown in Figure 2. It can seen that the compressive strength of concrete decreases as copper slag content increases as shown in Figure 2. The percentage reduction in compressive strength for 5%, 10%, 15% and 20% of copper slag was 2.9528, 6.1381, 23.6224 and 34.2013 respectively after 28 days of curing. It can be seen that the reduction in compressive strength is minor up to 10% of copper slag replacement butbeyond 10% of copper slag, there is significant reduction in compressive strength due to the increase in free water content in the mixes.

4.3 X- RAY DIFFRACTION

The XRD patterns of mix C0, C10 and C20 are shown in Figure 3, Figure 4 and Figure 5 respectively. The XRD patterns are present in the form of diffracted peaks of different hydration products. The

intensity of these diffracted peaks is plotted against the diffraction angle of 2θ (degrees). The intensity of diffracted peaks was measured in counts per second (cps). From Figure 3 to 5 it can be observed that all the mixes consist of the peaks of qrartz, portlandite and alite on 2θ scale. But the portlandite is the main hydration product during the hydration of cement. The peaks of portlandite in all mixes represent the degree of hydration of cement. The major peaks of portlandite were observed at 18.1° and 34.1°. The peak of portlandite at 18.1° was overlapped with the peak of alite. The peak of portlandite at 34.1° was the highest among all the peaks of portlandite. The portlandite peak of mix C0 was showed the highest intensity of 1029.0 (cps) at 34.1° in Figure 3. The mixes C10 and C20 shows the intensity of 947.0 (cps) and 738.0 (cps) in Figures 4 and Figure 5 respectively. The mixes C0 and C10 were having almost similar diffracted peaks of portlandite, but the intensity of portlandite peak in mix C20 was very less as compared to mixes C0 and C10. It indicates that the degree of hydration of mixes C0 and mix C10 were quite similar, but the degree of hydration in mix C20 was less than mixes C0 and C10. It is concluded that 10% of copper slag slightly reduce the hydration of cement, but 20% of copper slag significantly reduces the hydration of cement.

5. CONCLUSIONS

The following conclusions are drawn from this investigation:

i) The workability of concrete increases as CS content increases. For 20% of CS, the workability increases by 8.21%.

ii) The compressive strength of concrete decreases as CS content increases for all curing ages. The reduction in compressive strength is minor up to 10% of CS but beyond 10% of CS, there is significant reduction in compressive strength due to the increase in free water content in mixes.

iii) XRD showed that the degree of hydration of mixes C0 and mix C10 were quite similar, but the degree of hydration in mix C20 was less than mixes C0 and C10. The10% of copper slag slightly reduces the hydration of cement, but 20% of copper slag significantly reduces the hydration of cement.

iv) Copper slag can be utilized as supplementary cement replacement material in concrete. The optimum content of CS as replacement of cement is recommended as 10%

Tables Table 1: Properties of OPC 43 grade cement

Characteristics	Value Obtained experimentally	Values specified by BIS 8112
Specific Gravity	3.15	-
Standard consistency	32%	-
Initial Setting time	146 minutes	30 minutes (min)
Final Setting time	244 minutes	600 minutes (max)
Compressive Strength		
3 days	24.60 N/mm^2	23 N/mm^2
7 days	35.87 N/mm^2	33 N/mm^2
28 days	48.45 N/mm^2	43 N/mm^2

Table 2: Physical properties of coarse aggregate and fine aggregate

Property	Fine aggregate	Coarse aggregate
Specific Gravity	2.64	2.6
Fineness modulus	2.71	6.71
Water absorption %	0.5	1

Table 3: Physical and chemical properties of copper slag

Physical properties	Copper slag
Particle Shape	Irregular
Appearance	Black & glassy
Type	Air cooled
Specific gravity	3.51
Bulk density(g/cm^3)	1.9-2.4
Hardness	6-7mohs
Chemical component	**% of Chemical component**
SiO_2	28%
Fe_2O_3	57.50%
Al_2O_3	4%
CaO	2.50%
MgO	1.20%

Table 4: Mix proportions of concrete mixes

Mixes	Cement (Kg/m^3)	Copper slag (Kg/m^3)	Water (L/m^3)	w/c ratio	Coarse aggregates (Kg/m^3)	Fine aggregates (Kg/m^3)	Compacting factor
C0	432	0	186	0.43	1167	548	0.849
C5	410.4	21.6	186	0.43	1167	548	0.865
C10	388.8	43.2	186	0.43	1167	548	0.871
C15	367.2	64.8	186	0.43	1167	548	0.9
C20	345.6	86.4	186	0.43	1167	548	0.925

Table 5: Test results for compressive strength of concrete

Compressive strength (N/mm^2)				
Mixes	**3 days**	**7 days**	**14 days**	**28 days**
C0	24.15	30.85	37.5	43.01
C5	22.35	30.6	35.45	41.74
C10	21.49	28.98	33.95	40.37
C15	16.9	23.34	27.11	32.85
C20	14.19	18.5	22.87	28.3

FIGURES

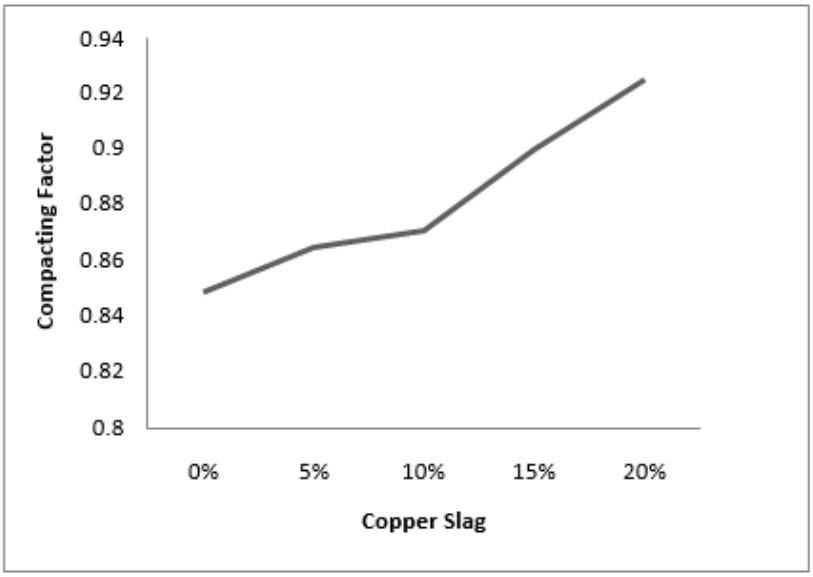

Figure 1: Compacting factor of concrete with different replacement levels of copper slag

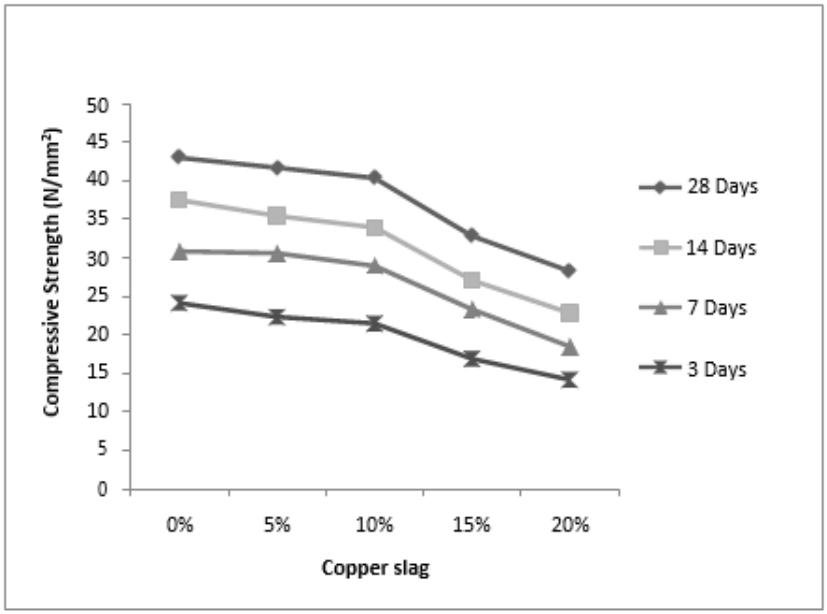

Figure 2: Compressive strength of concrete with different replacement levels of copper slag

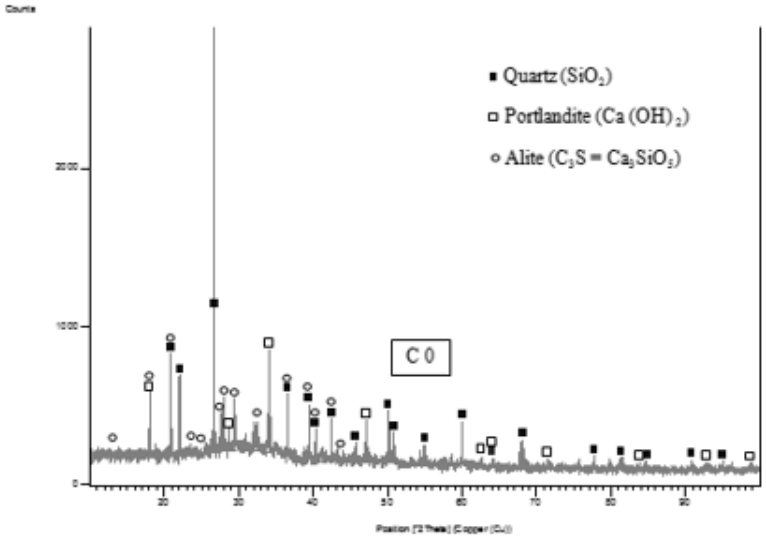

Figure 3: X-ray diffraction pattern of mix C0

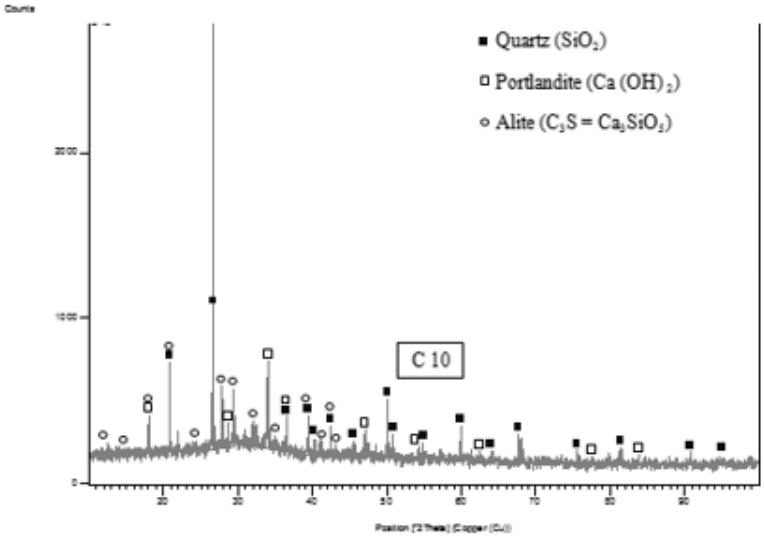

Figure 4: X-ray diffraction pattern of mix C10

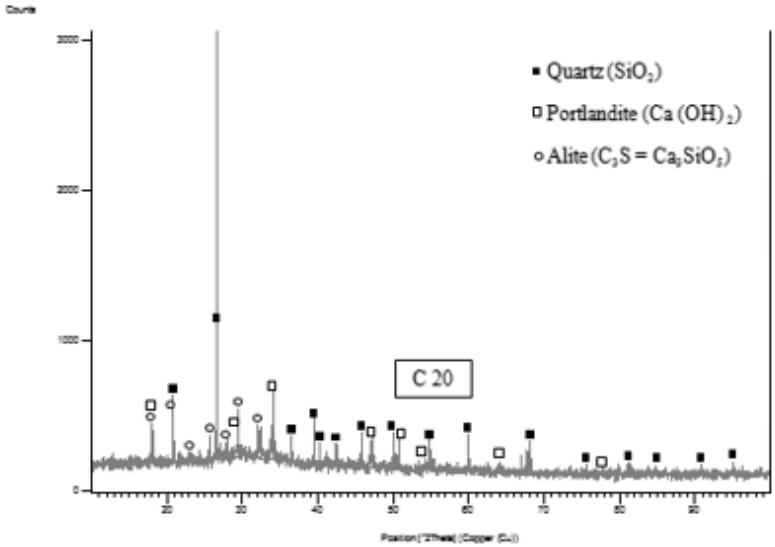

Figure 5: X-ray diffraction pattern of mix C20

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Determination Of Calcium And Magnesium In Clinker, Cement & Fly Ash Based Cement By EDTA Without Using Masking Reagents

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A B S T R A C T

This method for determination of calcium and magnesium in clinker, ordinary Portland cement and fly ash based Portland pozzolana cement, is unique among the other available methods (such as gravimetric method, titrimetric method by using KMnO4/ EDTA method as per IS:4032- [1].It is very quick method as compared to above. The uniqueness of this method is that it gives accurate results in short interval of time and also avoids use of high temperature muffle furnace. Calcium and magnesium are determined in filtrate after separation of combined ferric oxide and alumina from the sample solution.

Key words: CaO, MgO, Cement, Clinker & EDTA.

INTRODUCTION:

Cement including Ordinary Portland cement, Portland Pozzolana cement are used as binding materials of concrete in civil construction industries. There is huge requirement of cement in construction work all over the world. The main ingredient of concrete structure as binder of concrete is cement. The quality of cement as per standard specification is very important for the cement manufacturing industries as well as for the civil construction industries for making solid and long life structure. Cement consists of mainly following constituents.

CHEMICAL COMPOSITION OF ORDINARY PORTLAND CEMENT, (OPC)[2]:

TABLE-1

Sr. No.	Constituents	Range
1	Silica (as SiO_2)	17-25 %
2	Calcium (as CaO)	60-67%
3	Alumina (as Al_2O_3)	3-8 %
4	Iron (as Fe_2O_3)	0.5-6%
5	Magnesia (as MgO)	0.5-4.0 %
6	Oxides of alkalis (Na_2O & K_2O)	0.3-1.2 %
7	Sulphuric Anhydride (as SO_3)	2.0 -3.5 %

Above range of constituents exists in complex compound form.

Sr.No.	Formal Name of Compound	Abbreviated Formula #	Formula	Brogue's Equation for calculating %age of compound
1	Tri Calcium Silicate	C_3S	(3CaO. SiO_2)	4.07*CaO-7.60*SiO_2-6.72*Al_2O_3-1.43*Fe2O3-2.85*SO3
2	Di Calcium Silicate	C_2S	(2CaO.SiO_2)	2.87*SiO_2-0.75*C_3S
3	Tri Calcium Aluminate	C_3A	(3CaO. Al_2O_3)	2.65*Al_2O_3-1.69*Fe_2O_3
4	Tetra Calcium Aluminate Ferrite	C_4AF	(4CaO. Al_2O_3.Fe_2O_3)	3.04*Fe_2O_3

#The above symbols are used in cement manufacturing industries.

The main constituent of the cement and clinker is calcium oxide (CaO) which is the major factor for cement quality. It is determined by several analytical techniques. One of the analytical technique to determine calcium oxide is complexometric titration with EDTA. It isalso used to find the total calcium and magnesium content of milk, sea water and various solid materials. It can also be used to determine the total hardness of fresh water provided the solutions used are diluted. The combined concentration of calcium and magnesium ions is the total hardness of water.

The method uses a very large molecule called EDTA which forms a complex with calcium and magnesium ions. A blue dye called Eriochrome Black T (EBT) is used as the indicator. This blue dye also forms a complex with the calcium and magnesium ions, changing colour from blue to pink in the process. The dye–metal ion complex is less stable than the EDTA–metal ion complex. For the titration, the sample solution containing the calcium and magnesium ion reacts with an excess of EDTA. The

indicator is added and colour changes to blue as all the Ca2+ and Mg2+ ions present are complexed with the EDTA.

The main reaction is:

$Ca^{2+} + EDTA^{4-} \rightarrow [Ca\text{-}EDTA]^{2-}$

In complexometric titration with EDTA, interferences are mainly caused from cations of iron, aluminium and manganese. Apart from reacting with EDTA, these metals also react irreversevely with indicators. These cations also give rise to colour change in the indicator, making difficult to detect the end point. However, there are standard test methods available to determine calcium oxide and magnesium oxide by EDTA method. But the present study has been carried out to develop a method quicker, accurate and less expensive method.

GLASS APPARATUS REQUIRED:

Pipette (25 ml,10ml & 50ml capacity),burette(10 ml, 25 ml capacity), Volumetric flask (100ml, 250ml, 500ml & 1000ml capacity), Conical flask-100 ml capacity.

CHEMICAL & REAGENTS REQUIRED:

STANDARD EDTA SOLUTION:0.01 M:[3]

Dissolve 1.8612 gm of disodium ethylenediamine tetra acetate dihydrate in 200 ml hot water and make up the volume to 500 ml in calibrated volumetric flask.

BUFFER SOLUTION-pH 10: [4]

Dissolve 70 g of ammonium chloride in 570 ml of ammonium hydroxide (sp gr. 0.90) and make up volume to 1000 ml with distilled water in a calibrated volumetric flask.

STANDARD ZINC SOLUTION:0.01M: [5]

Dissolve accurately weighed 0.6537gm of granulated zinc in minimum quantity of dilute hydrochloric acid (1:1). Make up to mark with distilled water in a calibrated volumetric flask of capacity 1000 ml.

ERICHROME BLACK T- INDICATOR: [6]

Grind 100 mg of indicator with 10 gm of sodium chloride till homogeneous mixture is obtained and store in an airtight container.

PATTON-REEDERS INDICATOR /(P & R) INDICATOR :[7]

[2-hydroxy-1-(2-hydroxy-4 - Sulpho -1-Napthylazo)-3-Napthoic Acid], ($C_{21}H_{14}N_2O_7S$)

Grind 100 mg of indicator with 10 gm of sodium or potassium sulphate till homogeneous mixture is obtained and store in airtight bottle.

METHYLTHYMOLBLUE INDICATOR-MIXTURE:

0.1 gm Methylthymolblue mixed with 10 gm of KNO_3.

STANDARDIZATION OF EDTA- SOLUTION USING STANDARD ZINC SOLUTION:

Take 10 ml of standard zinc solution in conical flask. Add 20 ml buffer solution of pH-10 and warm at 50 to 60^{o} C. Add 50 mg Erichrome Black –T indicator and titrate with 0.01M EDTA till the color changes from red wine to clear pink blue. Note the volume of EDTA used and calculate the molarity of EDTA by using formula, M1xV1 = M2xV2

Note: Use burette of least count 0.05 ml for accurate results.

TABLE - 2

Sr. No.	Molarity of zinc Solution (M1)	Volume of Zinc Solution (V1-ml)	Molarity of EDTA Solution (M2)	Volume of EDTA Solution (V2-ml)	M1 x V1 = M2 x V2 M2 = (M1 x V1) / V2
1	0.01	10	To be determined	9.85	0.01015
2	0.01	10	To be determined	9.9	0.0101
3	0.01	10	To be determined	9.85	0.01015
Average Molarity of EDTA Solution using standard zinc solution:					**0.01013**

STANDARDIZATION OF EDTA- SOLUTION USING CERTIFIED REFERENCE MATERIAL $CaCO_3$:

Weigh known quantity (W-gm) of the standard $CaCO_3$ traceable to NIST into a 100 ml capacity conical flask and dissolve in 25 ml- distilled water and add 10 ml of 10 % KOH solution and shake well to adjust pH to highly alkaline range of 12 or slightly more. Add approximately 50 ml of distilled water and 50 mg of solid P & R indicator. Titrate against 0.01 M EDTA solution to a sharp change in colour from wine red to clear blue.

TABLE-3

Purity of $CaCO_3$ [8]	A	=	99.98%
Equivalent weight of $CaCO_3$	B	=	50.03
Molecular weight of $CaCO_3$	C	=	100.09
Purity Fraction	D	=	Purity/100

TABLE-4

Sr. No.	Weight of $CaCO_3$ Taken (W- gm)	Purity fraction of $CaCO_3$ (D)	Volume of EDTA Consumed (V-ml)	Molecular weight of $CaCO_3$ (C)	Molarity of EDTA Solution (W*D*1000)/(V*C)
I	0.01232	0.9998	12.1	100.09	0.01017
II	0.01626	0.9998	15.9	100.09	0.01021
III	0.01142	0.9998	11.2	100.09	0.01018
Average Molarity of EDTA Solution using standard Calcium Carbonate					**0.01019**
Final Average Molarity used for Calculation M-EDTA Average of (molarity standardized against Zinc and molarity standardized					**0.01016**

PREPARATION OF SOLUTION OF CEMENT/CLINKER SAMPLE:

Take approximately0.5 gm of the sample in an evaporating clean dish / clean beaker; moisten with 10 ml of water at room temperature. Care should be taken to avoid any lump formation. Add 5 to 10 ml of HCl and agitate with the help of policeman (glass rod fitted with rubber / glass rod soften at the agitating end by melting), till completely dissolves. Evaporate the solution till complete dryness on a steam bath or hot plate, temperature of the steam bath or hot plate should be adjusted in such a way to avoid any spurting of the sample from dish or beaker. Without heating the residue, further treat it 1:1 HCl and water (20-ml) and digest on the water bath or hot plate. Dilute the volume with equal volume of hot water. Filter through ash less filter paper (What man No 40 or its equivalent), wash the separated silica thoroughly with hot water and reserve the residue. Re evaporate the filtrate to dryness; bake the residue in an oven for one hour at 105 to 110 deg. C. Treat the residue with 20-ml of HCl (1:1) and heat the

bath or on hot plate. Dilute the solution with an equal volume of hot water and catch the silica into the filter paper. Make up the filtrate up to 250 ml in a calibrated volumetric flask. Reserve the filtrate and washings for separation of combined aluminium and Iron oxide.

SEPARATION OF COMBINED FERRIC OXIDE AND ALUMINA FROM SAMPLE SOLUTION:

From the filtrate reserved above in 250ml volumetric flask, take 50 ml aliquot into a conical flask, add few drops (1-ml of H_2O_2 + 1-ml of HNO_3) and heat to boiling in order to oxidize any ferrous iron to the ferric condition. Treat the boiled solution with (1:1) NH_4OH drop- wise till colour of the solution becomes distinctly yellow and subsequently treat this solution with 0.5 gm of $NH_4S_2O_8$ and pour excess four –five drops NH4OH until the indicator turns yellow. Boil the solution for further one minute and allow the precipitate to settle for five minutes and filter through Whatman No. 41filter paper and wash with 2 % hot NH_4NO_3.

Transfer precipitate&filter paper to original beaker and dissolve the precipitate with hot dilute HCL (1:3) and dilute to 100 ml. Re-precipitate the hydroxides and filter the solution. Wash the precipitate with two to 10 ml portion of the hot NH4NO3 solution. Combine the filtrate and washings and make up to 250-ml in a calibrated volumetric flask. Reserve the filtrate for determination of CaO and MgO.

(Final Solution:0.5 gm into 250 ml volumetric flask, Aliquot 50 ml and volume make up 250 ml)

DETERMINATION OF CALCIUM BY EDTA-METHOD:

Take 50-ml aliquot of the solution (reserved for determination of CaO & MgO) into a conical flask and add 1 gm of NH_4NO_3+ 20 ml of 10 % KOH and shake well to adjust pH to highly alkaline range of 12 or slightly more. Add approximately 50 ml of distilled water and 50 mg of P&R indicator. Titrate against 0.01 M EDTA solution to a sharp change in colour from wine red to clear blue. Record the volume of EDTA consumed (V-ml).

Calculations: Calculate the percentage of CaO

% CaO= (V-EDTA x 56.08 x M-EDTA x100) / (1000 x Weight of samplein aliquot taken)

Where,

V-ml = Volume of EDTA solution consumed-ml,

M-EDTA = Molarity of EDTA

DETERMINATION OF MAGNESIUM BY EDTA-METHOD:

Take 50- ml of the solution (reserved for determination of CaO and MgO) add 1-gm of NH_4NO_3 + 20 ml. of buffer solution of pH 10 and 20 ml of NH4OH. Add 50 mg of methylthymolblue indicator and titrate against 0.01-M EDTA to change colour from light blue to colourless greyor50-mg of EBT indicator and titrate it against standard 0.01-M EDTA solution until the colour changes from pink to light green. This titration gives the sum of the calcium and magnesium oxide present in the sample solution. Titre value of magnesium is obtained by subtracting the titre value of calcium oxide (V-ml) from the total titre valueof (calcium + magnesium oxide), (V1ml).

Calculations:

Calculate the percentage of MgO

Magnesium Oxide (MgO) percent = (V1-V) x 40.32 x M-EDTA x 100 / (1000 x W) Where,

V1 = Titre value used for total (calcium + magnesium)
V = Titre value used for Calcium
W = Weight of the sample in aliquot.

VALIDATION OF METHOD USING REFERENCE MATERIAL OF KNOWN CHEMICAL COMPOSITION FOR CaO AND MgO:

NAME OF REFERENCE MATERIAL FOR ORDINARY PORTLAND CEMENT(OPC):

Reference Material CRM-1012K-Ordinary Portland Cement Standard [9]

TABLE-5 CHEMICAL COMPOSITION OF CRM-1012K

Sr. No.	Chemical Constituents	% By Mass	Expanded Uncertainty (Coverage Factor k=2)
1	Loss On Ignition	3.56	± 0.02
2	Silica (as SiO_2)	21.27	± 0.06
3	Iron (as Fe_2O_3)	4.14	± 0.02
4	Alumina (as Al_2O_3)	4.51	± 0.05
5	Calcium (as CaO)	61.42	± 0.14
6	Magnesium (as MgO)	1.21	± 0.04
7	Sulphuric Anhydride (SO_3)	2.06	± 0.03
8	Sodium (as Na_2O)	0.22	± 0.003
9	Potassium (as K_2O)	0.46	± 0.006

NAME OF REFERENCE MATERIAL FOR PORTLAND POZZOLANA CEMENT:

Reference Material CRM-1016C- Portland Pozzolana Cement Standard [10]

TABLE - 6 CHEMICAL COMPOSITION OF CRM-1016C

Sr. No.	Chemical Constituents	% By Mass	Expanded Uncertainty (Coverage Factor k=2)
1	Loss On Ignition	4.58	± 0.02
2	Silica (as SiO_2)	31.24	± 0.11
3	Iron (as Fe_2O_3)	3.78	± 0.03
4	Alumina (as Al_2O_3)	9.99	± 0.05
5	Calcium (as CaO)	44.89	± 0.12
6	Magnesium (as MgO)	1.13	± 0.04
7	Sulphuric Anhydride (SO_3)	2.18	± 0.03
8	Sodium (as Na_2O)	0.27	± 0.01
9	Potassium (as K_2O)	0.6	± 0.01

EXPERIMENT CARRIED OUT : TABLE-7

Sample Name	Sr. No.	Weight of sampletaken (gm)	Volume make Up (ml)	Aliquot takenfor R_2O_3 Separation(ml)	Volume make from the filtrate of R_2O_3 (ml)	Aliquottakenfor testing of CaO & MgO(ml)	Final weightof aliquot(samplein aliquot)(gm)	EDTAUsed forCaO Titration(V-ml)	EDTAUsed forMgO Titration(V1-ml)	% CaO = (V*M*56.08*100)/ (wt. of aliquot*1000)	% MgO = (V1-V)*M*40.32* 100) /(wt. of aliquot* 1000)
ORDINARY PORTLAND CEMENT	1	0.5034	250	50	250	50	0.02014	21.7	22.3	61.4	1.22
	2	0.5031	250	50	250	50	0.02012	21.7	22.3	61.44	1.22
	3	0.5012	250	50	250	50	0.02005	21.6	22.2	61.39	1.23
	Average analytical results of Calcium Oxide (CaO) and Magnesium Oxide (MgO) %									**61.41**	**1.22**
PORTLAND POZZOLANA	1	0.5018	250	50	250	50	0.02007	15.8	16.4	44.85	1.22
	2	0.5042	250	50	250	50	0.02017	15.9	16.4	44.92	1.02
	3	0.5024	250	50	250	50	0.0201	15.8	16.4	44.8	1.22
	Average analytical results of Calcium Oxide (CaO) and Magnesium Oxide (MgO) %									**44.86**	**1.15**

(PRIMARY DATA ON ACTUAL ANALYSIS BASIS) [11]

TABLE - 8 COMPARISON OF ANALYTICAL VALUES WITH KNOWN VALUES

Analyte		CRM-1012K (Ordinary Portland Cement)			CRM-1016C (Portland Pozzolana Cement)		
		Analytical Results by experiment	CRM Value	Remarks	Analytical Results by experiment	CRM Value	Remarks
Calcium (as CaO)	I	61.4	**61.42 ± 0.14**	Average analytical value of CaO is within the specified limit of CRM.	44.85	**44.89 ± 0.12**	Average analytical value of CaO is within the specified limit of CRM.
	II	61.44			44.92		
	III	61.39			44.8		
Average Value		**61.41**			**44.86**		
Magnesium (as MgO)	I	1.22	**1.21 ± 0.04**	Average analytical value of MgO is within the specified limit of CRM.	1.22	**1.13 ± 0.04**	Average analytical value of MgO is within the specified limit of CRM.
	II	1.22			1.02		
	III	1.23			1.22		
Average Value		**1.22**			**1.15**		

CONCLUSION:

The above test procedure for determination of Calcium Oxide (CaO) and Magnesium Oxide (MgO) has been used for determination of known values of two Certified Reference Materials (CRM). The analytical results by experiments have been found within the specified tolerance of the known values.

REFERENCE:

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2. Safari Books Online (Chemical Composition of Portland Cement) 3. Clause 4.1.15 of IS 4032-1985
4. Clause 4.1.13 of IS 4032-19855. Clause 4.1.14 of IS 4032-1985
6. Clause 4.1.16 of IS 4032-1985
7. Clause 4.1.18 of IS 4032-1985
8. Certified Reference Material (CRM) traceable to NIST of CaCO3
9. Certificate of Analysis for Ordinary Portland Cement (OPC) Standard (CRM-1012 K)
10. Certificate of Analysis for Portland Pozzolana Cement (PPC) Standard (CRM-1016 C)
11. Primary Analytical Data for OPC and PPC.

Retention Of Posts Luted With Resin Cement In Canals Obturated Using A Eugenol Sealer -An Invitro Study

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ABSTRACT

Objectives: *to examine the effect post-obturation sequencing had on the retention of prefabricated posts luted with a resin cement into canals previously obturated using a eugenol based sealer.*

Materials and methods*: 64 single rooted upper anterior teeth were decoronated , and root canals were filed, cleaned and shaped with gatesglidden drills and stainless steel hand k-files. Teeth were then divided into 4 groups of 16 specimens each. Group 1 was not obturated and served as a control. The other 3 groups were obturated with gutta-percha and a eugenol based sealer. Post space preparation and post cementation were completed at 3 different post obturation intervals – immediate(Group 2) , 1 week(Group 3), and 4 weks(Group 4). Ten mm deep post spaces were prepared with peso reamers and prefabricated posts were cemented with Rely-X-Arc cement. Following 48 hours of storage,specimens were mounted in metal tubes with acrylic and posts were removed in tensile mode using an Instron testing machine at 1mm/min with data recorded in kgs.*

Results: *using 1-way ANOVA and bonferroni tests,Group 1 demonstrated significantly greater mean retention strength values than Group 2 and 3($P<0.05$) which in turn had significantly greater mean retention strength values than Group 4($P<0.05$).*

Clinical significance and conclusion: *post space preparation and post cementation with resin cement should not be significantly delayed following obturation when a eugenol containing sealer has been used. Additionally, removal of some canal wall dentin beyond the periphery of the obturated canal is recommended.*

Key words: Prefabricated posts , Resin Cement, Eugenol Sealer, Tensile Bond Strength, Mean Retention Strength Values

INTRODUCTION:

Modern endodontic therapy has provided dentistry with the ability to retain teeth that would have been extracted without hesitation few decades ago.[1] With proper endodontic treatment and an adequate post endodontic restoration, pulpless teeth can serve indefinitely as an integral part of the dental apparatus.

Once the endodontic treatment has been completed these teeth which are already weakened due to caries, previous restoration, fracture, endodontic access opening and instrumentation need to be adequately restored.[1,2,3] Weine[4] claimed that "more endodontically treated teeth are lost due to poor post endodontic restoration". Swartz, Skidmore and Griffin[5] found that "failure rate of endodontically treated teeth was almost double in cases without adequate post endodontic restoration".

The post endodontic restoration very often includes the placement of a post in the root canal to provide support to the core which replaces the missing coronal tooth structure. Current literature suggests that the post helps to retain the core and distribute the forces of mastication evenly to the root, periodontal ligament and surrounding bone[1].

A large variety of post designs have been described in the literature. They may be custom- made or prefabricated. Prefabricated posts allow fast and easy techniques to be used in the restoration of endodontically treated teeth. These posts are still the preferred choice for many because of their retention values and high strength. They are stronger and have different surface designs for added retention[6,7]. There are basically two types of prefabricated posts that are currently available: Active and passive. . Irrespective of their retentive qualities, all posts require a luting cement to seal the irregularities between the post and the canal walls.

The need for cementation of posts was first recognized by Fauchard[7] in 1742 when he recommended the use of specifically formulated mastic compound[7]. Traditionally, zinc- phosphate , zinc-polycarboxylate and glass ionomer cements have been used to lute posts. Advances in dental material sciences produced resin cements which were shown to provide micro mechanical and chemical bonding to both dentin and metal[8]. Studies have reported that retention strength afforded by the resin cements is 150-200% more than zinc- phosphate and glass ionomer cements[9].Eugenol based sealers have remained the gold standard for endodontic obturation due to their various beneficial properties and long term clinical success[10,11]. However concern has been expressed that the radical scavenging properties of residual eugenol (2-methoxy-4- allyphenol) inhibits the polymerization of composites[12],thereby significantly reducing the bond strength of resin cements.

A vital point to note is the length of time eugenol containing sealers remain in contact with dentin prior to their removal for post space preparation. Several studies have reported that the release of eugenol from zinc oxide-eugenol sealers into the dentin is rapid during the first 24 hours, after which it decreases slowly over a period of time[13,14,15]

In order to improve bond strengths of posts luted with resin cements in canals obturated with eugenol sealer, it is important to identify the optimum time for post cementation when the residual eugenol in dentin is negligible enough to affect the polymerization of the composite resin. Therefore, the aim of the present study was to investigate the effect of three different post-obturation intervals (immediate, 1 week and 4 weeks) on the retention of posts luted with a resin cement into canals obturated using a eugenol based sealer.

MATERIALS AND METHODS:

Sixty four human single rooted maxillary anterior teeth that were freshly extracted due to gross caries involvement were selected for the study and stored in distilled water. The teeth were decoronated at the cemento enamel junction using carborundum disks rotating at slow speed in a micromotor straight hand piece. The coronal pulp tissue was removed and the root canal spaces were debrided manually using barbed broaches. Teeth deemed to have significantly smaller or larger root canal spaces were discarded to standardize the extent of dentin preparation as much as possible. A single operator performed all specimen preparation and post cementation. The canals were negotiated with sizes 10 and 15 stainless steel K-files until the tip of size 15 was observed to exit from the apical foramen. Working length was then established 0.5 mm short of this length. The coronal portion of each canal was shaped with sizes 2-6 gates glidden drills. The canals were then subsequently cleaned and shaped using successively larger stainless steel hand K-files till size 40. The size of master apical file was kept constant at 25. 1 ml of 3% NaOCl was introduced into the canals after every instrument using a 2 ml syringe. Smear layer was removed using 17% EDTA (Rc Prep) coated on each file during instrumentation of the canal.The canals were also recapitulated with a size 25 K-file to ensure patency of the canal terminus.

The specimens were randomly divided into 4 groups, each containing 16 teeth. Group 1 was not obturated and served as a control. Group 2, 3, and 4 were obturated with gutta-percha and a eugenol based sealer (Endoflas FS)

With the exception of no obturation for the controls (Group 1), all specimens were treated in the following sequence. A size 25 gutta-percha point was placed into the canal and fitted to the working

length to establish a tugback. The canals were then dried with paper points. The sealer was prepared and used according to the manufacturer's instructions. The master cone coated with sealer was placed twice to the working length, to ensure that the sealer coated the root canal walls adequately. Accessory cones were placed and warm lateral condensation in conjunction with vertical compaction was accomplished using stainless steel hand spreaders and pluggers respectively.

Post space preparation and post cementation were then completed at three different post- obturation intervals.

Group 2: Immediate (45-60 minutes following obturation)

Group 3: 1 week

Group 4: 4 weeks

Group 1 (Un-obturated controls) had post spaces prepared and posts cemented at 1 week. Specimens were then stored in 100% humidity at room temperature.

For post space preparation, the coronal 10mm of each root canal was instrumented with size 1-6 peeso reamers. The purpose of the canal preparation was to completely remove gutta-percha and sealer from the post space to establish a fresh dentin surface, and to provide an adequate dimension for resin cement around the post.

The canals were then rinsed with water and dried with paper points, followed by acid etching with 37% Orthophosphoric acid for 15 seconds. Following this the canals were rinsed and blot-dried to leave the dentin surface moist. Dentin bonding agent (single bond, 3MESPE)was then applied to the canal walls with a fine applicator tip and light cured for 20 seconds. Rely X-Arc resin (3M ESPE) cement was then dispensed onto the mixing pad and mixed according to the manufacturer's instructions. The surface of the posts as well as the canal space was coated with resin cement and the posts were manually inserted as close to the centre of the post space as possible to maintain an even film thickness of the cement circumferentially. Cement flash was removed with a probe and glycerin was placed over the exposed cement to facilitate setting. This was followed by light curing for 20 seconds. After this, the roots were gently notched using a carborundum disk and the specimens were mounted into 1 cm diameter metal tubes using acrylic. Dental surveyor was used in mounting the specimens to enable subsequent post removal in a direction parallel to the long axis of the posts.

FIG 1: Mounting the specimens with a surveyor to ensure parallelism

The specimens were secured and the posts were extracted using vise clamps mounted in a universal testing machine (Instron machine) operated in a tensile mode at 1mm/ min until the posts were dislodged from the canals. Data was recorded in kilograms and subsequently examined using ANOVA and Bonferroni tests.

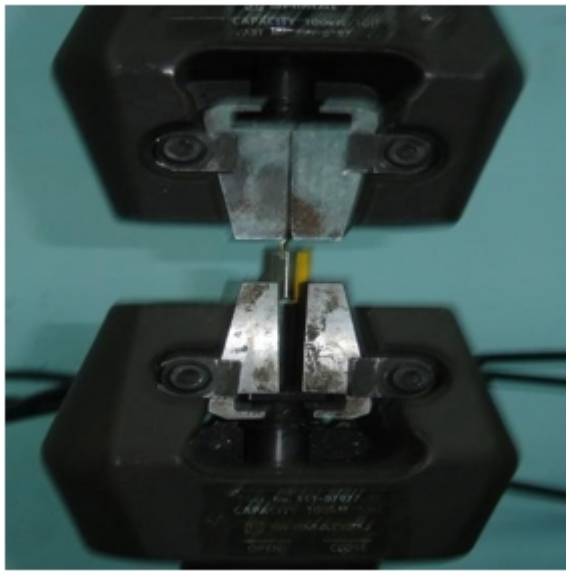

FIG 2: Post gripped using vise clamps in an Universal Instron testing machineRESULTS:

All the specimens were subjected to tensile force using an Instron universal testing machine and the load at which fracture occurred was recorded in kilograms (kgs) as shown in (Table 1)

Mean tensile bond strengths of all the groups tested along with the standard deviation. Analysis of variance technique (one-way ANOVA) was used to evaluate the difference among 4 groups. (Table 2)

Table 1: Retention strength values in Kgs

S. No.	Group 1	Group 2	Group 3	Group 4
1	59.31	38.6	43.38	20.62
2	60.23	36.32	41.8	19.63
3	61.42	38.73	43.15	18.82
4	58.76	39.41	42.76	21.34
5	63.68	37.05	40.26	19.37
6	61.26	38.46	41.86	18.61
7	62.8	39.24	36.62	25.84
8	61.06	37.27	39.98	18.62
9	64.71	36.86	42.43	19.47
10	58.67	39.02	44.64	18.38
11	61.63	38.8	40.18	19.13
12	60.08	33.03	39.62	20.36
13	62.94	38.62	43.29	18.43
14	61.76	39.14	42.86	19.49
15	59.85	38.44	42.73	17.92
16	63.3	37.19	41.93	18.92

Table 2: One-way ANOVA test

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	14007.9	3	4669.3	1416.79	0
Within Groups	197.74	60	3.29		
Total	14205.64	63			

* The mean difference is significant at the .05 level.

Normal plot of the data on tensile bond strength indicated that the observations had come from normal distribution. Hence, to carry out test of equality of mean tensile bond strength for the four groups, Post Hoc test with multiple comparisons using Bonferroni procedure was carried out with SPSS software.

The results showed that **Group 1** (unobturated controls) demonstrated highest mean retention strength values than groups 2, 3 and 4. **Group 3** (1 week) showed highest mean retention strength values among all the obturated groups tested. **Group 2** (immediate) showed significantly higher ($P<0.05$) mean retention strength values than Group 4 (4 weeks). Group 4 (4 weeks) had the lowest mean retention strength values than all groups. There were statistically significant differences between all groups but there was no significant differences in mean retention strength values between Group 2 (immediate) and Group 3 (1 week). **(Fig 3)**

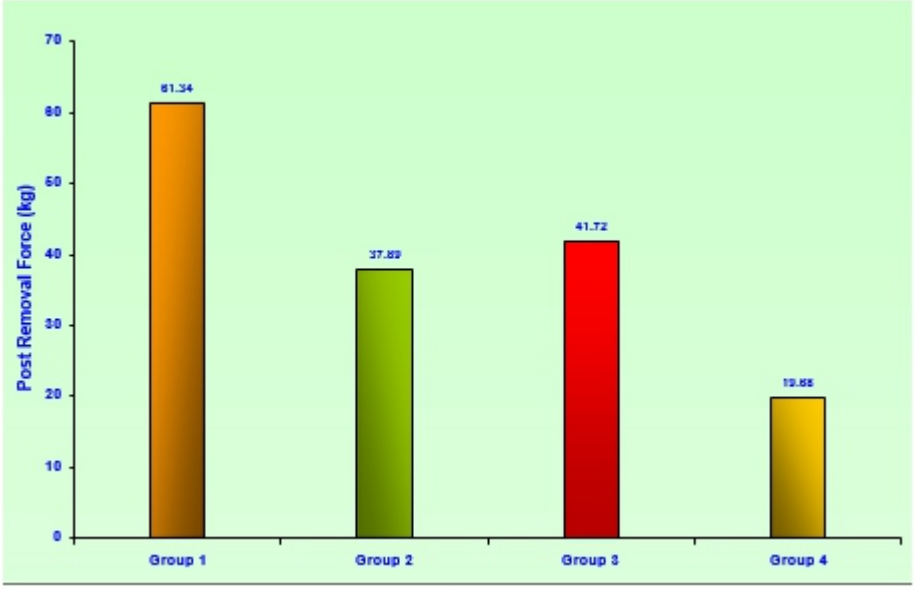

Figure 3: Comparison of mean retention strength values of all study groups

DISCUSSION:

The working hypothesis in this study is that either immediate or considerably delayed removal of eugenol based sealer might provide mean retention strength values equivalent to unobturated controls. However, both of these experimental premises were disproved as all of the obturated groups had lower mean retention strength values than the unobturated group. This finding implies that bond strengths to canal well dentin may have been compromised by residues of eugenol sealer despite removing sufficient eugenol contaminated dentin[10,16].

Specimens prepared immediately (Group 2) and at 1 week (Group 3) did not have significantly different mean retention strength values. These values compared favourably with the reports of Boone et al, who examined the effects of sequencing on post space preparation and cementation using eugenol and resin based sealers. They also reported no significant difference in post retention when the post space preparation was doneimmediately or after 1 week following obturation. The reason for this result could be because, the diffusion of eugenol immediately and after 1 week of obturation into the dentinal tubules would not have been considerable enough to affect the bond[10,16,17,18]Also post space preparation immediately and at 1 week might have atleast partially counteracted the eugenol diffusion as described by Hume[13].

However, specimens prepared at 4 weeks (Group 4) did have significantly lower mean retention strength values than the immediate and 1 week group. These findings are similar to those reported by Boone et al[19] who suggested that may be some mechanical removal of eugenol contaminated canal wall

necessary for optimal post retention. Though this was done in the present study, it did not improve bond strengths considerably. The significantly reduced mean retention strength values in Group 4 suggest that eugenol dispersion may have progressed considerably into the dentinal tubules well beyond the dentinal layer removed by the larger sized peeso drills so that the residual eugenol adversely affected the cement tooth bond[10,13,19].

In the present study, overall higher mean retention strength values were observed as compared to other reports[18,19,20,21]. One reason could be that in those studies, matched sizes of preparation drills and posts were employed. It could be that the close fit of the post to the canal walls may have reduced the film thickness of the resin cement, thus lowering the retention strengths. Whereas in our study, post spaces were prepared with size 6 peeso reamers with a diameter of 1.7mm, while the posts employed had a diameter of 1.5mm. This made the post passive and provided a uniform space for the resin cement.

Various methods have been suggested for increasing bond strengths of resin cements to negate the eugenol influence. These include Canal irrigation with ethyl alcohol[22], Etching the prepared post space with 37% H_3PO_4[23], Removing the smear layer prior to post cementation by using 17% EDTA with 5.25% NaOCl[24,25,26,27]. Of these methods, in the present study H3PO4 was used for etching the post space, as it is a part of bonding process. But this did not produce bond strengths as high as the unobturated controls.

On analyzing the results of this study, the ideal time for post space preparation and post cementation using resin cement in the presence of eugenol sealer is still unclear. In the present study, both immediate and delayed groups exhibited lesser retention values than the unobturated controls. This suggests that in both cases eugenol influence was stillpresent. Future studies could be directed towards identification of optimum time when residual eugenol may have absolutely no effect on the retention of posts by resin cement.

CONCLUSION:

This invitro study was undertaken to investigate the effect post obturation sequencing had on retention of endodontic posts luted with a resin cement into canals previously obturated using a eugenol based sealer.

The following conclusions can be drawn from the present study -

1. The complete absence of eugenol in the root canal will guarantee the highest possible bond strength

2. Whenever possible, post space preparation should be carried out within a week after obturation.

3. If delayed, it is better to wait more than 4 weeks to completely negate the influence of eugenol.

4. In any case, removal of sufficient amount of eugenol-impregnated dentin surrounding the root canal will enhance the bond strength.

5. Selecting a slightly undersized post allows for a uniform thickness of resin cement to develop adequate bond strength.

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A Review On Recycling Of Waste Glass In Construction Industry

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ABSTRACT

The utilization of many industrial byproducts in the construction industry is now well-developed as it helps in improving the sustainability in two ways. First, reuse of the materials which otherwise will burden the environment and will be occupying scarce land resource. Second, it minimizes the degradation of land and the environment as a result of comparatively less digging. Quantities of waste glass have been on the rise in recent years due to an increase in industrialization and the rapid improvement in the standard of living. Unfortunately, the majority of waste glass is not being recycled but rather abandoned, and is therefore the cause of certain serious problems such as the waste of natural resources and environmental pollution. Thus, efforts have been made during the last decade on exploring the possibility of reusing waste glass as a construction material. Waste glasses can be used as raw materials for cement production as siliceous sources. Ground glass powders exhibit very good pozzolanic reactivity and can be used as cement replacement. The crushed glasses can also use as aggregates for Portland cement concrete. The present paper reviewed the different studies regarding use of waste glass in concrete and finds their effect on the properties of concrete.

Keywords: Concrete, Recycling, Waste glass

INTRODUCTION

Glass in general is a highly transparent material formed by melting a mixture of materials such as silica, soda ash, and $CaCO_3$ at high temperatures followed by cooling during which solidification occurs without crystallization. Glass is widely used in our lives through manufactured products such as sheet glass, bottles, glassware, and vacuum tubing. Glass has been indispensable to man's life due to such properties as pliability to take any shape with ease, bright surface, resistance to abrasion, safety and durability. The utility ranges of the glass increase the amount of the waste glass (WG). The WG can be recycled into newglass. However, not all used glass can be recycled into newglass because of impurities,

cost or mixed colors. Nonrecyclable waste glass constitutes a problem for solid waste disposal in many municipalities. The current practice is still to landfill most of the nonrecyclable glass. Since the glass is not biodegradable, landfills do not provide an environment-friendly solution [1]. Consequently, there is a strong need to utilize waste glasses. Traditionally, most nonrecyclable mixed-color broken glasses are coming from the bottling industry. In addition, the recently initiated business of recycling mercury-containing fluorescent lamps also produces a large quantity of nonrecyclable waste glass. The fluorescent lamp recycling facility crushes the fluorescent lamps, separates the metal caps, and recovers mercury. For 55,000 tubes recycled, approximately 30 m3 of waste glass will be generated. In the future, with the environmental law being strictly enforced and with the increasing use of fluorescent lighting systems for energy efficiency, it is expected that more nonrecyclable waste glass will be accumulated from the fluorescent lamp recycling business [2]. Foreign countries have long been taking much effort to recycle waste glass bottles. A bottle recovery system, through which empty bottles previously containing alcoholic beverages, refreshing beverages, condiments, milk, etc. are collected, washed, and reused, has already been established. In addition, broken bottles and bottles previously containing chemicals, cosmetics, etc. are melted down to be reused or crushed and turned into paving material, block material, glass marble, glass tile, glass fiber, lightweight blowing agents, etc [3–4].

Use of recycled materials in construction is among the most attractive options because of the large quantity, low quality requirements and widespread sites of construction. The main applications include a partial replacement for aggregate in asphalt concrete and cement concrete. Recently, many studies have focused on the uses of wastes glassed as aggregates for cement concrete or as cement replacements [5-7]. The present paper reviewed the different applications of waste glass in construction industry.

PHYSICAL AND CHEMICAL PROPERTIES OF GLASSES

The more common types of silicate glasses are vitreous silica, soda-lime glasses, borosilicate glasses, lead glasses, barium glasses, and aluminosilicate glasses. Vitreous silica glass has very low thermal expansion, is very hard, and resists high temperatures (1000–1500 °C). It is also the most resistant against weathering. Soda-lime glass is transparent, easily formed and most suitable for window. It has a high thermal expansion and poor resistance to heat (500– 600 °C). Borosilicate glasses stand heat expansion much better than window glass. Used for chemical glassware, cooking glass, car head lamps, etc. Borosilicate glasses have as main constituent's silica and boron oxide. They have fairly low coefficients of thermal expansion is 3.25×10–6/°C as compared to about 9×10−6/°C for a typical soda-lime glass. The typical compositions of different types for different applications are listed in Table 1. In chemical compositions of soda-lime glass shows SiO_2 around 73% which is good for a pozolanic

material. Lead glass has also good amount of silica oxide, but due to the high lead content in the glass. This can be potentially leached into the environment. Borosilicate is not commonly used. Thus soda-lime glass can used in construction industry.

Table 1: Chemical composition of selected commercial glasses in percentage [8]

Glasses and uses	SiO_2	Al_2O_3	Na_2O	K_2O	MgO
Soda-lime glasses					
Containers	66-75	0.7-7	16-Dec	0.1-3	0.1-5
Float	73-74		13.5-15	0.2	3.6-3.8
Sheet	71-73	0.5-1.5	15-Dec		1.5-3.5
Light bulbs	73	1	17		4
Tempered ovenware	75	1.5	14		
Borosilicate					
Chemical apparatus	81	2	4		
Pharmaceutical	72	6	7	1	
Tungsten sealing	74	1	4		
Lead glasses					
Color TV funnel	54	2	4	9	
Neon tubing	63	1	8	6	
Electronic parts	56	2	4	9	
Optical dense flint	32		1	2	
Barium glasses					
Color TV panel	65	2	7	9	2
Optical dense barium crown	36	4			
Aluminosilicate glasses					
Combustion tubes	62	17	1		7
Fiberglass	64.5	24.5	0.5		10.5
Resistor substrates	57	16			7

USE OF WASTE GLASS IN CEMENT PRODUCTION

The waste glass can be used in the production of cement due to SiO^2 component in the glass. However the alkali component in the glass results increase in alkali in cement. It is well known that alkalis has adverse effect on the production of cement, but waste glass as araw material in cement production has no of advantages. Many researchers have work on the suitability of waste glass in cement production.

An innovative approach of using waste glass in cement production was proposed by Chen [9] in a laboratory and cement production plant. The laboratory characterization of 32 types of glass show that the chemical composition of glass does not vary significantly with its color or origin but depends on its application. The alkali content of glass, a major concern for cement production varies from 0 to 22%.

The SO_3 content of the clinker is comparable with that obtained without the loading of glass. The alkaline content shows a slight increase but still within three times the standard deviation obtained from the statistical data of the past year. The detailed analysis of the quality of the cement product shows that there is not any significant impact of glass for the feeding rate tested. In other study the effects of the glass in cement raw mix on clinker burning were investigated [10]. The experimental results show that the addition of the glass into cement raw mix results in the formation of more liquid phase between 950°C to 1250°C compared with conventional raw meals. Decreases C3S content in the clinker and increases NC_8A_3 content, which leads to flash setting and poor strength development of the cement. Therefore, it is necessary to increase the SG value [$SG=SO_3 100\ \%/(\ 1.292\ K_2O+0.85\ Na_2O)$] of the clinker when the glass is present in the raw mix.

USE OF WASTE GLASSES AS CEMENT CONCRETE AGGREGATE

The use of waste glass as aggregate in concrete has also been studied by many researchers. The deleterious alkali- silica reaction (ASR) has been a major concern with such concrete [11]. Meyer et al [12], report some of the possible measures available to mitigate ASR. These included grinding glasses to a particle size less than 300 lm, use of mineral admixtures, using alkali-resistant glass, sealing concrete to keep it dry or using low alkali cement. However Ahmad and Aimin [13] concluded that up to 50% of both fine and coarse aggregates could be replaced in concrete by glass aggregates with acceptable strength development. This finding is in general agreement with results obtained by Park et al [14]. Improvements in cullet production methods was shown by Sangha et al [15] to produce concrete that was stronger than that made with natural aggregate in tension and compression when using glass cullet up to replacement levels of 60%. The authors attributed the improved strength to better bonding between glass cullet and cement matrixthan that achieved with natural aggregate. The process used for producing recycled glass aggregate used in this study has been described elsewhere, Sangha et al [15]. The resulting cullet is free of sharp edges and all the paper, foil, plastic labels and organic impurities that may have been attached to the glass are liberated and separated during the implosion process. It was observed by the authors that the temperature of glass cullet after production remained high for the next 24 h. It was thus considered that concrete made with glass cullet could have significantly different thermal properties. Corinaldesi et al [16] investigated that mechanical properties and microstructures of mortars with 30–70% replacement of fine sand with ground glasses. It was noticed that no deleterious effect could be detected at a macroscopic level due to the reaction between cement paste and ground waste glass with particle size up to 100 micron. On the contrary, a strong improvement of the mortar mechanical performance was detected, due to the positive contribution of the waste glass to the micro-structural properties. The use of waste glasses as aggregates did not have a marked effect on the

workability of concrete, but decreased the slump, air content and fresh unit weight [17]. Concrete with glass aggregates would require a higher content of water than conventional aggregates to reach the same workability. The compressive, flexural and indirect tensile strengths as well as Schmidt hardness decrease in proportion to an increase in waste glass aggregates. The strength noticeably decreased when the glass content was more than 20% [17].

USE OF WASTE GLASS AS SUPPLEMENTARY CEMENT REPLACEMENT MATERIAL

Glass is amorphous and contains relatively large amounts of silicon and calcium. Thus it can be claimed that it is pozzolanic or even cementitious in nature, even when it is finely ground. Therefore, glass powder can be considered as a replacement for cement in concrete [18]. The pozzolanic properties of glass are noticeable at particle sizes below approximately 100 mm. Studies by Shi et al [19] showed that not only glass with particle size below 100 mm can have a pozzolanic reactivity but also its effect is greater than fly ash at low level of cement replacement (10-20%). Work by Chen et al [20] revealed that a glass powder with particle size less than 75 mm possessed cementitious capability and improves compressive strength, resistance to sulphate attack and chloride ion penetration, for replacing of cement up to 50%. Idir et al [21] indicated that the pozzolanic activity has a tendency to enhance with finer glass powder. Equivalent or superior compressive strength was attained when using up to 40% of mixed-colour glass powder with a particle size less than 40 mm when compared with control specimens. Shao et al [22] measured strength of the lime–glass mixtures as the pozzolanic index for three glass powders. The strength results indicated that the 38 μm glass satisfied the minimum strength requirement at 7-day test, and attained an increase in strength after additional 21 days of curing in water. The strength of the mixture with 150 μm glass was far below the limit because the size of the glass was too coarse to serve as a pozzolan. The 75-mm glass performed marginally. Its 7-day strength was slightly lower than the threshold value, while its additional 21-day curing in water enhanced the strength to a satisfactory level. Nishikawa et al [23] was found that the strength of cement paste at 90 days increased with the glass content increase up to 25%. The Blaine fineness of the glass powder is about 400m2/kg, but no chemical analysis of the glass powder was given. Dyer and Dhir [24] measured the compressive strength development of cement pastes containing white, green, and amber cullet. There is clearly some difference in the strength development of mortars containing different-colored finely ground glass cullets: white and green produce a slight increase in 28-day compressive strengths relative to the glass-free control at replacement levels of around 10%, whereas amber finely ground glass cullet merely achieves similar strengths to the control. The rate of strength gain in mortars containing finely ground glass cullet is noticeably higher between 7 and 28 days compared to the control. This behavior implies

CONCLUSION

After reviewed the different studies, uses of waste glass in concrete are summarized as follows:

- Waste glasses cans be used as raw materials for cement production as siliceous sources.

- Waste glass containing relatively large quantities of silicon and calcium, and posses' pozzolanic and cementitious nature when it is finely ground. Thus, it can be used as a cement replacement in Portland cement concrete. As expected, its pozzolanic reactivity increases as its finenesses increase.

- The use of crushed glasses as aggregates for Portland cement concrete does have some negative effect on properties of the concrete; however, practicle applicability can still be produced even using 100% crushed glass as aggregates

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