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## **OPTIMISING THE STRENGTH PERFORMANCE OF FLY ASH AND BASIC OXYGEN SLAG MIXTURES USING GYPSUM**

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number of words in your main text, followed by the number of tables and illustrations. **3117 words and 7 tables**

## **ABSTRACT**

The lower environmental impact and economic cost of cements made from waste materials makes them an attractive alternative to Portland cement. This research was undertaken to study the performance of cement made by sulphate activation of a Basic Oxygen Slag (BOS) and Run of Station Ash (ROSA) blended with Red-gypsum (RG) and Plaster Board Gypsum (PG). Two-component and three-component mixes were tested as part of a systematic process of optimising for strength. All mixes had similar water to binder (w/b) ratio 0.3. The results showed PG and RG can be used with sulphate to form a sulphate activated pozzolan BOS-ROSA binder. Sample solutions were collected from a high pressure through flow test and analysed using Inductive Coupled Plasma to measure the risk of leaching of heavy metals.

Keywords: Slag, Compressive strength, Run of Station ash, Gypsum, By-product

## 1. Introduction

It has become increasingly apparent that, in order to achieve the best performance when replacing all, or almost all of the Portland cement in concrete with secondary minerals, it is necessary to use more than one replacement material (Claisse *et al.* 2010). In the work reported in this paper, all of the cement was replaced with combinations of ash, slag and gypsum. In order to achieve the maximum possible environmental and economic benefit, the materials which were selected for use had the lowest possible market value. Thus Run Of Station Ash (ROSA) was used in preference to the Pulverised Fuel Ash (PFA) that is normally used in concrete because, although from the same source, the ROSA is not classified or processed in any way and thus has no market value. Similarly Basic Oxygen Slag (BOS), which is a steel slag, was used in preference to Granulated Blastfurnace Slag because it has lower economic value. Also the two sources of gypsum were both waste streams: “Red Gypsum” (RG) was obtained from a titanium dioxide (white pigment) plant and waste Plasterboard Gypsum (PG) was obtained from construction site off-cuts and production rejects.

The aim of this work was to test combinations of these materials and identify mixes that gave good strength and were thus potential candidates for commercial use. When working with several materials in multiple combinations and proportions, the numbers of combinations become far too large for a “full matrix” factorial experimental design in which all possible mixes are tested.

A total of 378 samples with binary combinations of materials and 738 with ternary combinations were tested to give a good indication of the properties of different combinations. Artificial Neural Networks and Response Surface analysis were used to build predictive models, but these proved to be too sensitive to minor changes in the source materials which caused complete failure of the models (Karami, 2009). Thus this paper reports on a simpler approach in which binary combinations were tested and then used to guide the choice of ternary mixes to make.

## 2. Literature Review.

The materials which were used in this project are not in current use in significant amounts in concrete production. There are, however, reports of their use in previous research.

Jones and McCarthy (2005) investigated the potential of using unprocessed Run of Station Ash (low lime) from bituminous coal combustion in foamed concrete. They used coarse fly ash as a replacement for sand in concrete. They reported that using run of station ash had a significant beneficial effect on the 28 day strength of foamed concrete.

Manjit and Singh (1995) researched the setting time, compressive strength, and water absorption of a gypsum anhydrite- slag pastes. Their results showed the compressive strength of these mixes increased with curing time. The results also showed the combination of gypsum and slag with a small amount of other chemical activators achieved higher compressive strength.

Kourounis *et al.* (2007) reported that cement replacement using steel slag developed lower strength at all ages compared to Portland cement, and the strength of the cementitious pastes decreased with increasing slag content. The slag-cements required less water to give the same workability with the same binder content and had increased setting times.

Xuequan *et al.* (1999) replaced 50% of their cement with steel slag and fly ash. Their mix had a compressive strength 57.1 MPa after 28 days which was higher than the pure cement control mix. They also found that drying shrinkage of the new mix was much lower than the control at all ages.

Shih, Wu *et al.* (2004) used waste steel slag for making bricks. They found that if an appropriate amount of steel slag (less than 10%) was added to the mixture, the firing temperature could be reduced. The compressive strength and shrinkage reduced and the water absorption increased with increasing the slag content.

Ganjian, *et al.* (2007) investigated of the optimisation the compressive strength of Basic Oxygen Slag- cement Bypass Dust - Plasterboard Gypsum combinations and developed a semi dry mix with the strength of 11 MPa (w/c =0.3) and 31 MPa(w/c=0.13) at 28 days. They used the novel binder as 100% cement replacement for road sub base construction and subgrade stabilisation in two site trials.

White (2006) investigated the stabilisation of Class C fly ash with calcium activators such as hydrated lime and Cement Kiln Dust (CKD) in the construction of a structural pavement base layer. He found strength and freeze-thaw durability of the stabilised mix increased. Hydrated fly ash has been used in pavement and sub-grade and base construction projects because of its low unit weight, high strength, ease of compaction, economical benefits and abundant supply.

Ambarish and Chillara (2007) studied class F fly ash and its modification with lime or gypsum. Their result showed that adding up to 10% of lime added to class F fly ash improved the shear strength. Adding 0.5, 5 and 15 of gypsum to the lime modified mix caused it to gain progressively higher strength in the early curing period but if no gypsum was used it required 45 days or more to gain considerable shear strength.

The summary of the previous researches which reviewed in this paper are listed in Table 1.

**Table 1. Summary of other researches in cement replacement**

Materials	Mixture type	w/c	Maximum 28 days compressive strength(MPa)/curing condition	Industrial application of the product	References
Fly ash (class F),lime(10%),gypsum(1%)	paste	31.5 - 35.5%	3.2/soaked	Road and embankment construction,waste containment liners,cut off walls and vertical barriers	Ambarish and Chillara (2007)
Slag (5%) and Clay(95%)	paste	3/5	11±0.6/heated 1100°C	brick	Shih, Wu et al. (2004)
Clinker (45%), high-alumina cement(2%), Steel slag(30%) and fly ash(20%)	mortar	c:s:w= 1:2.5:0.5	57.1	Portland Cement replacement	Xuequan et al. (1999)
Steel Slag (15%), OPC (85%)	paste	26%	55	Portland Cement replacement	Kourounis et al. (2007)
Phosphogypsum anhydrite(50%) , granulated blast furnace slag(50%) and activators	paste	Not specified	33	Building materials, boards,tiles, artificial amrble, masonry	Manjit and Singh (1995)
OPC(300kg/m <sup>3</sup> ),Coarse Flyash(633kg/m <sup>3</sup> ) and foam(7.2kg/m <sup>3</sup> )	concrete	50%	2	Portland Cement replacement	Jones and McCarthy (2005)
plasterboard-derived recycled gypsum (15%), By Pass Dust (5%) and Basic Oxygen Slag (80%)	<u>paste</u>	<u>30%</u>	<u>11</u>	Portland Cement replacement in road base and subbase (Cemented Base Materials)	Ganjian, et al. (2007).

### 3. Research objective

The objective of this research was to evaluate and optimise the effect of two different types of waste gypsum on the compressive strength of paste samples with BOS-ROSA binders. The mixes are intended for use in concrete for low strength applications such as road bases, house foundations and mine and trench backfill.

### 4. Experimental Methods

#### 4.1. Materials

Basic Oxygen Slag (BOS) was obtained from Corus UK Scunthorpe plant. BOS is a by-product from steel making in an oxygen blast furnace and is obtained directly from the plant without granulation. When this material is used for applications such as road embankments it is weathered to hydrate the calcium oxide. The material used in this project was not weathered but it was ground to pass a 600 $\mu$ m sieve. The fineness and particle size distribution have significant influence on the strength of cementing material (Zhao *et al.* ,2007)

Ground waste plasterboard gypsum (PG) was obtained from Lafarge Plasterboard in Bristol UK. This material comes from off-cuts from site and production rejects from the manufacturing plant. It is processed at a recycling plant where 99% of the paper is removed; however, it still contained minor amounts of glass and paper. The plasterboard was also ground to pass a 600 $\mu$ m sieve.

ROSA was obtained from Ratcliffe-on-Soar Power Station, UK. It is understood that this ash came from burning a conventional UK bituminous coal. It was not classified and was therefore not a designated PFA.

**Table 2 Chemical oxides composition of the starting materials used (in weight %) obtained using X-ray fluorescence measurements.**

Sample	BOS	ROSA	PG	RG
SiO <sub>2</sub>	11.43	45.91	2.43	12.71
TiO <sub>2</sub>	0.39	1.41	0.03	0.39
Al <sub>2</sub> O <sub>3</sub>	1.60	26.51	0.81	1.72
Fe <sub>2</sub> O <sub>3</sub>	28.24	5.23	0.36	21.26
MnO	4.35	0.08	0.00	2.66
MgO	8.27	2.13	0.40	6.18
CaO	41.29	6.88	37.30	35.89
Na <sub>2</sub> O	0.02	0.61	0.03	0.00
K <sub>2</sub> O	0.02	1.35	0.24	0.02
P <sub>2</sub> O <sub>5</sub>	1.48	0.98	0.02	1.62
SO <sub>3</sub>	0.44	1.37	53.07	9.31
LOI	1.12	7.11	4.09	7.1

Red Gypsum (RG) was obtained from Huntsman Tioxide, UK. This is a titanogypsum from titanium dioxide production. This process produces both pure white gypsum and the red gypsum

which acquires its colour from iron oxide impurities. The white gypsum is sold for plasterboard production but the red gypsum is normally landfilled.

The chemical compositions of the materials used are presented in Table 2.

#### *4.2. Experimental Design*

The mixes were optimised in two steps:

In step 1 binary (2 component) mixes were tested and optimised for compressive strength.

In step 2 a third material was added to the optimised mix and the ternary blend was then optimised.

#### *4.3. Mixing and Casting*

All mixes used a water/binder ratio of 0.3 to keep consistency in all pastes and as a minimum w/c ratio to be able to compact all samples. Mixing was carried out in a 2-litre capacity mixer. The mixing time was 5 minutes for all mixes (two minutes dry binder mixing, 1 minute while adding water and 2 minutes more).

The flow of the pastes was measured by a modified flow table (BS 4551 and ASTM C230) The diameter of paste spread was measured for each mix.

Pastes were cast in 50 mm cubes and fully compacted with a vibrating table in three layers. After casting, the moulds were kept in the laboratory environment for 24 hours. At this age, most specimens were de-moulded and cured at  $20\pm 1$  °C and 98% RH until 3, 7 and 28 days. The compressive strength was calculated as the average of three specimens.

### **5. Results and Discussion**

#### *5.1. Step 1 – Binary Mixes.*

##### *5.1.1. Compressive Strength*

In step 1 the effect of mixing PG, and RG with BOS and ROSA was studied separately. PG and RG acted as sulphate activators for BOS and ROSA. The mixes were made with the two raw materials and water. The results of the compressive strength of samples from this step are shown in Fig. 1 to 5 and helped in designing the ternary mixes. The standard errors within replicate sample groups were within 3%.

The compressive strength of all BOS-ROSA specimens increased with time as expected. BOS by itself has very low compressive strength when mixed with water. The hydration process is similar to that of cement but the rate is lower. Wang and Yan (2001) observed that steel slag can be regarded as a cementitious material with low activity.

Fig. 1 shows by adding 10% of ROSA increased the compressive strength of the mix to more than 300% of that of BOS by itself at each age. The optimum amount of ROSA mixed with BOS was 10%. Increasing ROSA from 10 to 60% caused a dramatic drop in compressive strength at 28 days. However increasing the ROSA percentage from 60% to 90% by weight in the mixes resulted higher compressive strength at each age. This was probably due to the increased silica and alumina contents in mixes as a result of increasing the ROSA.

Red Gypsum showed more reactivity with BOS than ROSA. The optimum amount of RG mixed with BOS was 20% at 28 days. However the mix with 40% RG had the highest compressive strength at 7 days. This might be due to availability of a rapidly reacting cementitious component (C<sub>4</sub>AF) in RG that gave higher strength at early ages. Increasing the RG content to more than 20% in all mixtures created a sticky mix with low compressive strength. Fig. 2 shows changes of compressive strength with BOS content.

PG was considered to be a sulphate activator for BOS. Almost all the PG-BOS pastes had more compressive strength than the RG-BOS mixes. At 20% addition PG shows more reactivity with BOS than RG (Fig. 2 and 3).

Both waste gypsums showed pozzolanic reactivity with ROSA.

PG is more effective at increasing compressive strength than RG with ROSA (Fig. 4 and 5). This may be because of the higher amount of sulphate and calcium oxide in PG than RG.

The compressive strength of all mixes in step 1 increased with time (Fig. 1-5).

### 5.1.2. Flow

The flow of all mixes increased with increasing BOS content (Table 3).

RG, PG and ROSA absorbed water in the mixing process by making the paste very dry and hard but BOS did not and the paste was almost liquid during mixing. After 3 minutes, ROSA released the absorbed water into the mix which resulted in more flow. Both PG and RG caused higher flow. RG absorbed more water and decreased the flow compared to the mix with the same amount of PG.

### 5.2. Step 2 – Ternary Mixes

In this step, a similar series of mixes were selected for BOS-ROSA combined with RG and PG. From the conclusions of previous research on compressive strength optimisation of basic oxygen slag and plasterboard gypsum waste (Sadeghi Pouya *et al.* 2007), it was known that the compressive strength of ternary mixes did not change in a linear way. Therefore the mixes were selected from optimum amount of BOS-ROSA ratio to gain higher compressive strength.

**Table 3. Flow of Binary mixes**

Materials		Flow mm
<b>BOS%</b>	<b>RG%</b>	
20	80	No Flow*
40	60	No Flow
60	40	95
80	20	121
100	0	High Flow**
<b>BOS%</b>	<b>PG%</b>	
20	80	No Flow
40	60	81
60	40	102
80	20	158
100	0	High Flow
<b>ROSA%</b>	<b>RG%</b>	
60	40	82
80	20	83
90	10	85
100	0	No Flow
<b>ROSA%</b>	<b>PG%</b>	
60	40	84
80	20	89
90	10	87
100	0	No Flow
<b>ROSA%</b>	<b>BOS%</b>	
0	100	No Flow
10	90	No Flow
20	80	80
40	60	116
60	40	162
80	20	High Flow
90	10	High Flow
100	0	High Flow

\*No Flow= Dry mix

\*\*High Flow=Flow more than 180mm

### 5.2.1. Compressive Strength

The results of this step are shown in Fig. 6 and 7.

The BOS-ROSA mixes with PG or RG resulted in higher compressive strength than that of binary mixes of BOS-ROSA at all ages. PG and RG acted as activators for both ROSA and BOS.

The final setting time and strength gain of these cements are attributed to the ettringite and hydrated alumina together with hydrated calcium silicate (Hewlett, 2001).

Fig. 6 illustrates the contours for compressive strength of ternary systems of ROSA-BOS-PG at 7 and 28 days. Increasing proportions of ROSA while using 10-15% PG and the rest BOS increased the strength. At 7 days, the compressive strength increased with increasing ROSA content to a peak at 75% ROSA. The maximum strength was for the mixes containing 55-65% ROSA. The

behaviour of ROSA-BOS-RG was slightly different to the ROSA-BOS-PG binders at 7 and 28 days.(Fig. 7). The maximum 7-day compressive strength was for 40-50% ROSA. The optimum amount of PG and RG was 10% with BOS-ROSA. Both RG and PG acted as activators for ROSA and BOS. The maximum compressive strength of ternary BOS-ROSA-PG or RG mixes was for a mix containing 30%BOS, 60%ROSA and 10% PG or RG. The compressive strength in all samples in step 2 increased with time. However some mixes had slightly more compressive strength at early ages and did not develop any pozzolanic reactions after 28 days.

### Flow

A small modified flow table was used for this experiment. Paste was poured in a 78mm diameter 70mm high cylinder in 3 layers and compacted with 10 tamps using a small rod (5mm diameter). After compaction the table was jolted 5 times and the diameter of the mixture was measured to show the flow. Flows of pastes with PG were higher than with RG. (Tables 4 and 5)

**Table 4. Compressive strength and flow of BOS-ROSA-RG mixes**

Materials			28 D Compressive Strength MPa	Density Kg/m <sup>3</sup>	Flow min
BOS%	ROSA%	RG%			
76	19	5	6.7	2175	High Flow
75	15	10	9.7	2160	High Flow
64	20	16	11.8	2095	146
50	45	5	13.8	1990	135
48	40	12	17.6	1987	120
40	30	30	17.9	1952	115
40	50	10	19.3	1920	105
40	45	15	20.2	1935	110
35	45	20	15.4	1900	102
30	60	10	18.9	1845	87
20	72	8	12.5	1788	85
15	80	5	10.2	1748	84

The flow of pastes increased by increasing the BOS content in both RG and PG pastes especially in BOS-ROSA-PG pastes. RG pastes had higher compressive strength than PG pastes in most combinations.

**Table 5. Compressive strength and flow of BOS-ROSA-PG mixes**

Materials			28 D Compressive Strength MPa	Density Kg/m <sup>3</sup>	Flow min
BOS%	ROSA%	PG%			
76	19	5	8.9	2188	High Flow
75	15	10	5.9	2182	High Flow
64	20	16	11.5	2133	High Flow
50	45	5	13.4	1990	136
48	40	12	7.1	2015	153
40	30	30	10.6	2006	119
40	50	10	18.8	1955	110

40	45	15	19.2	1979	112
35	45	20	15.5	1945	130
30	60	10	18.6	1883	95
20	72	8	13.4	1800	90
15	80	5	9.1	1768	85

Table 4 and 5 show that the flow of pastes increased by increasing the BOS content in both RG and PG pastes especially in BOS-ROSA-PG pastes. RG pastes had higher compressive strength than PG pastes in most combinations.

## 6. Permeability (High Pressure Flow Test) and ICP

Samples of the selected pastes were made using cylindrical moulds for a high pressure flow test in which water was pumped through the samples at very high pressures (Ganjian *et al.*, 2006). The specimens were cylindrical with 54 mm diameter and about 30 mm thickness. The specimens were cured for 28 days before testing. The time was taken to complete five selected sample volume hydraulic permeability tests was about 34 h for the 19.3 MPa strength BOS/ROSA/RG paste and it took 42hrs for the OPC sample.

The coefficient of permeability and the leaching were measured. Table 6 presents the results of permeability test.

An OPC sample was made as a control. The w/c in all the novel mixes was 0.23, to increase the compressive strength, and it was 0.5 in OPC mix to achieve sufficient flow to obtain a sample for leachate analysis..

Leaching values were obtained by analysis of the permeability tests.

**Table 6- Permeability of samples**

Mix	28 D Compressive Strength (MPa)	Coefficient of Permeability
OPC Sample	38	6.03E-10
48%BOS-40%ROSA-12%RG	17.6	6.22E-10
40%BOS-50%ROSA-10%RG	19.3	7.72E-10
30%BOS-60%ROSA-10%RG	18.9	6.91E-10
40%BOS-50%ROSA-10%PG	18.8	6.28E-10
30%BOS-60%ROSA-10%PG	18.6	6.90E-10

Results from the permeability test showed pastes with RG had greater coefficient of permeability than PG pastes with the same amount of BOS-ROSA.

All pastes had greater coefficients of permeability than the OPC sample.

Sample solutions were collected from the specimens tested and analysed using Inductive Coupled Plasma (ICP). The results of chemical analysis of solutions are presented in table 7.

**Table 7. ICP analysis of solution from 5 selected mixes**

Element	48BOS- 40ROSA- 12RG	40BOS- 50ROSA- 10RG	40BOS- 50ROSA- 10PG	30BOS- 60ROSA- 10RG	30BOS- 60ROSA- 10PG	Control Mix OPC
Na	297.6	23.41	47.15	245.2	115.3	43.3
Mg	<1	<1	<1	<1	<1	0
Al	0	0	0	0	0	0
Ba	0	0	0	0	0	0
Ca	276	589.52	522	598.9	560.2	154
K	448.9	140.50	110.60	398.8	230.6	35.1
Cr	0	0	0	0	0	0
Ni	0	0	0	0	0	0
Pb	0	0	0	0	0	0
S	137.1	127.38	124.8	184	159	14.5
Si	27.29	3.807	5.762	6.322	3.487	0
Sr	0	0	0	0	0	0
P	0	0	0	0	0	<0.2

From Table 7, it can be seen that no heavy metal elements leached from samples. The concentration of Potassium was higher than OPC in all samples that could be because of  $K_2O$  in PG and RG.

## 7. Discussion

Based on the results of the experiments in this research the hydration of slag started after mixing with water. Free lime from the ROSA and released ions from the slag and gypsum entered the solution.  $Ca^{2+}$ ,  $Al^{3+}$ ,  $Si^{4+}$ ,  $Mg^{2+}$ ,  $SO_4^{2-}$ ,  $Na^+$ ,  $K^+$  and  $OH^-$  ions were available in the system in early stage of hydration. The pH of the pore solution was measured in 30 minutes of the mixing which was from 10.8 to 12.2. As a result of hydration,  $Ca^{2+}$ ,  $Al^{3+}$ ,  $Si^{4+}$  and  $OH^-$  ions were partly removed from the pore solution. The sulphate content of the pore solution increased in order to balance the positive charges on the alkali metal ( $Na^+$ ,  $K^+$ ) and alkaline earth ( $Ca^{2+}$ ,  $Mg^{2+}$ ) ions created by the removal of the hydroxide ions. The hydration reactions accelerate the slag corrosion which results the increase in hydration.

The results confirm the observations from the review that blends of secondary minerals can be used to replace cement for low strength concrete applications. The results show that a characteristic strength of 15MPa is achievable with these mixes, which is sufficient for many footings and road foundations as well as the lower strength applications such as trench and mine backfill. While it was possible to make some general observations about the nature of the activation process, the high level of impurities (particularly in the red gypsum) makes it very difficult to draw specific conclusions. This should not be a barrier to commercial use, provided testing is carried out to confirm the suitability of the mixes for specific applications and procedures are in place to deal with the variability of the materials.

## 8. Conclusions

1. By-product cementitious materials such as BOS-RORSA-RG/PG combinations can be used at up to 100% cement replacement to make low compressive strength pastes. It is indicated that these pastes could be used to make low strength concretes
2. RG and PG can be used with BOS and ROSA as a source of sulphate and create sulphate activated binders with pozzolanic characteristics without using Portland cement.
3. The optimum mix ratio for BOS/ ROSA/PG and BOS/ROSA/RG was 30/60/10.
4. The 28 days compressive strength of ROSA/PG and ROSA /RG binders were increased by increasing the ROSA content in the binary mixes.
5. The compressive strength of the pastes increased with time, as with Portland cement specimens.
6. Mixes with PG had more flow than similar mixes with RG; this could be because of the higher solubility of PG compare to RG.
7. The coefficient of permeability of all binders was higher than the standard OPC sample.
8. There was no risk of leaching heavy metals from the pastes using BOS/ROSA/RG or PG.

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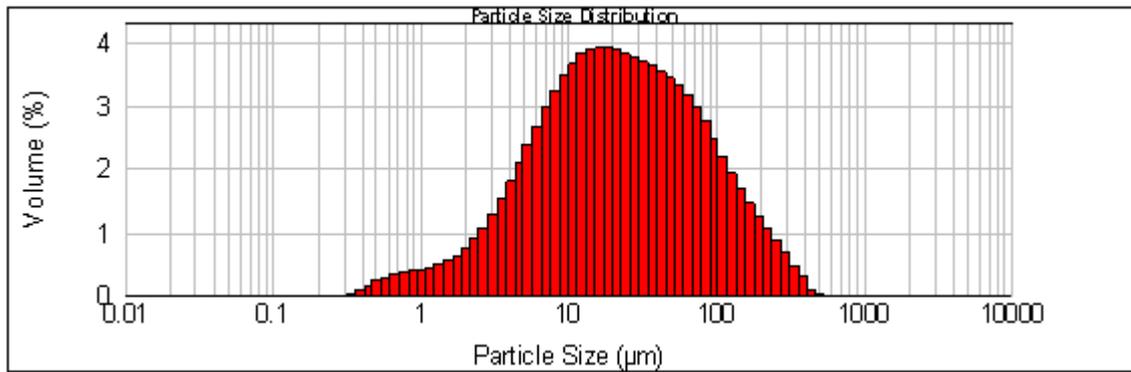


Figure1 Particle size analysis of ROSA (Ratcliff power station)

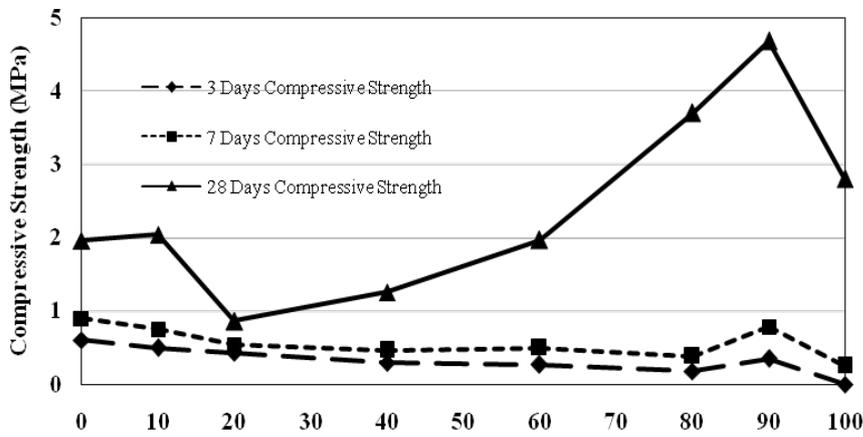


Figure 2 Compressive Strength of BOS-ROSA mixes at different ages

BOS %

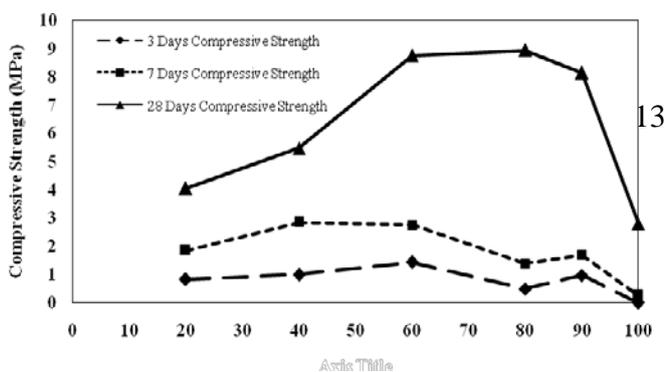


Figure 3. Compressive Strength of BOS-RG mixes in different ages

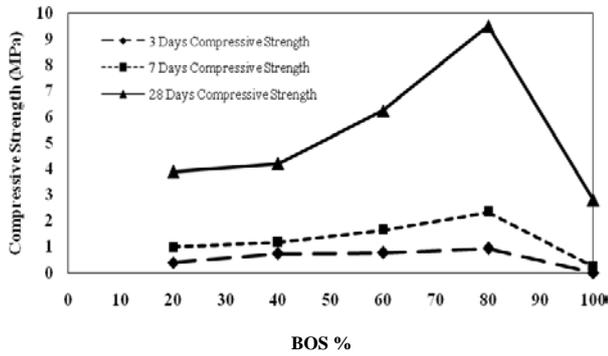


Figure 4. Compressive Strength of BOS-PG mixes in different ages

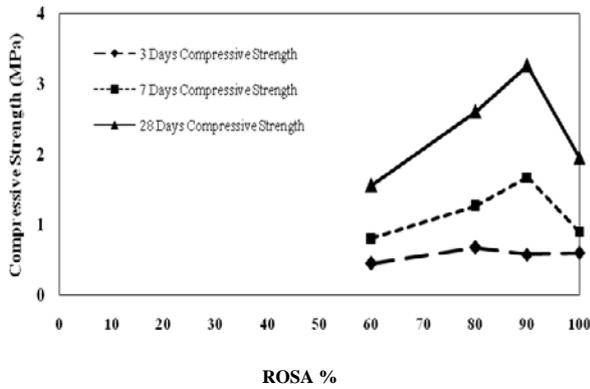


Figure 5. Compressive Strength of ROSA-RG mixes in different ages

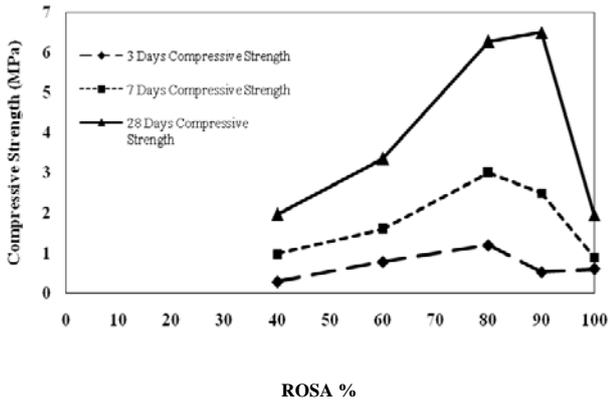


Figure 6. Compressive Strength of ROSA-PG mixes in different ages

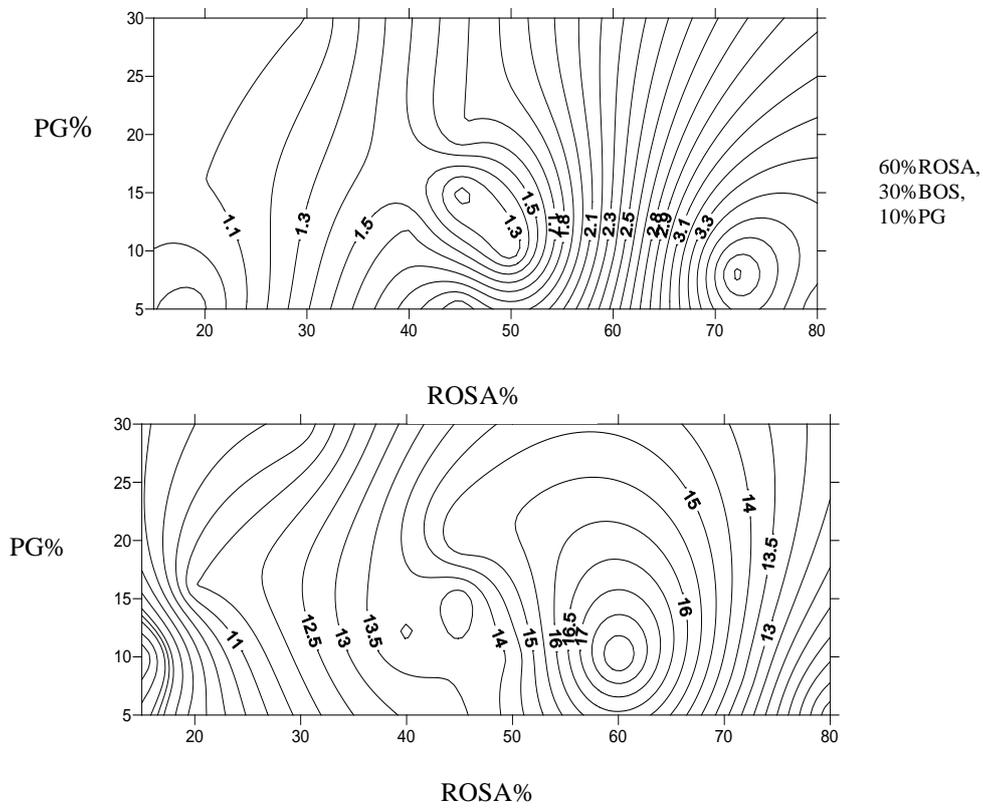


Figure 7. Compressive Strength (MPa) of BOS- ROSA-PG mixes (7days) and (28days)

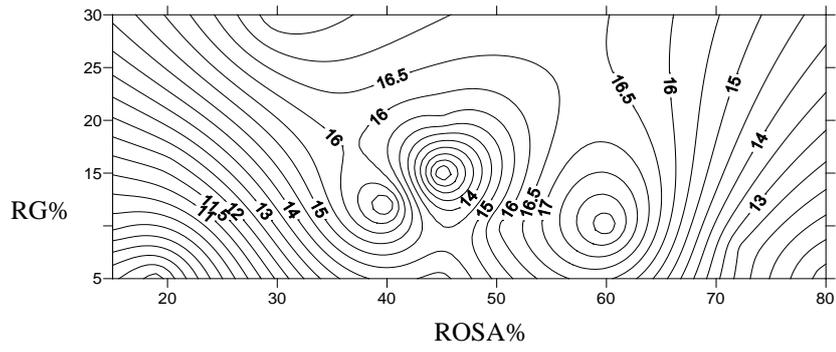
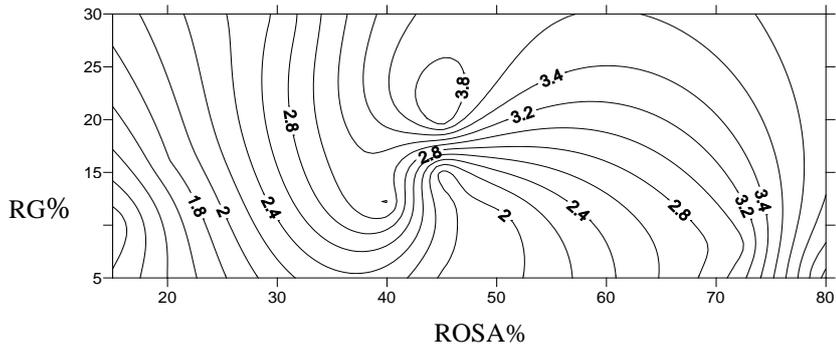


Figure 8. Compressive Strength (MPa) of BOS- ROSA-RG mixes (7days) and (28days)