

Study on Effectiveness of Processed and Unprocessed Black Liquor pulps in improving the properties of PPC mortar, Concrete and SCC

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Abstract: The cost of construction materials is increasing day by day because of high demand, scarcity of raw materials and high price of energy. From the view point of energy saving and over consumption of resources, the use of alternative constituents in construction materials is now a global concern. From this, the extensive research and development works towards exploring new ingredients are required for producing sustainable and environment friendly construction materials. Bagasse pulp liquor is one such material that can be used as a chemical admixture which is obtained as a by-product of paper manufacturing process. Around 5 million tons of bagasse pulp is obtained throughout the world each year. Since the material is a waste product from paper industry, this can be changed as an admixture by its effective use in concrete. In the present investigation black pulp liquor is added to fresh concrete in different dosages, the concrete is then tested for workability, compressive strength, flexural, split tensile strength and setting time. From results it is shown that 1% replacement of water with black pulp liquor increases the fresh properties of the concrete, 2% replacement of water with black pulp liquor increases the mechanical properties of the concrete and acts as a set retarder.

Keywords: Black pulp liquor, Super plasticizers, Portland pozzolana cement, SCC

1 Introduction

Waste management is a serious challenge for mankind. A simple solution is the proper utilization of this waste. Since concrete has become a highly consumed product, its vast usage has become uneconomical. Hence replacement of traditional materials by these by-products is a good alternative. Admixture is defined as a material other than cement, water and aggregate that is used as an ingredient of concrete and is added to the batch immediately before or during mixing (Shetty). Chemical admixtures are of growing importance in today's construction industry due to their ability to modify the properties of concrete. Though there are many commercially available chemical admixtures, it is eco-friendly and cheaper to use a by-product as a chemical admixture. India has the fastest growing market for paper globally and it presents an exciting scenario, paper consumption facilitates the economic growth and is estimated to touch 15 million tons by 2017-18. The mills use a variety of raw materials like wood, bamboo, recycled fiber, bagasse, wheat straw, rice husk, etc. Pulp is a lignocellulose fibrous material.

Pulp is a by-product produced from paper mill, have chemical constituents similar to established chemical admixtures and hence they have potential to act as an admixture. Kraft process and soda process are the two major alkaline processes used to convert the wood to pulp (Cardoso et al 2008) and the solution obtained after the removal of the fiber product is called black liquor. This liquid consists of large amount of dissolved lignin (Ayhan Demirbas, 2002). There are different methods available for extraction of lignin from black liquor but the conventional method like chemical pulping processes require large quantity of toxic materials and time requirement for the process is more. Nguyen Dang et al (2012) has developed new simple and efficient method for extraction of lignin using non-toxic



aluminum potassium sulfate dodecahydrate. Time requirement for extraction is also less in this method than the conventional method. Depending up on the type of raw material used and operational conditions of the pulping stages the chemical composition of the black liquor pulp will vary. This black liquor pulp consists of both organic and inorganic materials. This chemical composition has predominant role in determining its rheological and physical properties of liquor (Cardoso et al 2009). Darweesh et al (2013) studied the effectiveness of black liquor pulp as admixture in cement paste made up of using Ordinary Portland Cement (OPC) and Portland limestone cement (LPC). From the study it is concluded that black liquor pulp can be used as an accelerator because it reduces the setting time. It is also observed that there is increment in the compressive strength by the addition of black liquor pulp as admixture in cement paste. Modification in the microstructure especially the morphology and crystallinity of the formed hydrated product is also observed in this study. Mekkawi et al (2011) studied the effectiveness of black liquor pulp derived from rice straw pulp in concrete. Improvement in the workability, compaction and reduction in the setting time is observed in the concrete due to the addition of black liquor pulp. Effectiveness of extracted lignin from black pulp in improving the workability and compressive strength was studied by Kamoun et al (2003) and it is found that effectiveness of extracted lignin is comparable to those of a commercial LS-based plasticizer.

The use of super plasticizer (SP) has become common in the production of concrete with high workability and in most of the important projects across the world. SPs are high range water reducers. For a constant workability, high range water reducers can reduce water content up to 40%. SPs can be classified into following categories, Sulphonated melamine formaldehyde (SMF), Sulphonated naphthalene formaldehyde (SNF) and Lignosulphates (LS) (Shetty, Nevilee, Ramachandra, Metha and monterio). This entire sulphonated polymer based SPs were developed three decades back. The polycarboxylic ether based SP falls in the new generation SPs with a superior performance to that of conventional plasticizers and they are known as third generation SPs. LS are generally considered as first generation SPs and the Sulphonated formaldehyde condensates are called second generation SPs. LS, SNF and SMF based Sps work on the mechanism electrostatic repulsion whereas PCE cause dispersion of cement grains by steric hindrance.

Portland Pozzolana Cement (PPC) is used instead of ordinary Portland cement (OPC) because of its cost effectiveness, increased strength, durability and reduced emission of CO₂ in manufacture. Pozzolanic materials which are silicious /aluminous in nature by itself possess no cementitious properties. The commonly used pozzolanic materials are calcinated clay, fly ash and silica fumes. Pozzolanic material reacts with calcium hydroxide in the presence of water and forms CSH gel which has self cementitious property. This way the amount of soluble calcium hydroxide present in the matrix is reduced and it improves the durability by modifying the pore size distribution and thereby reducing the ingress of aggressive chemicals and water. Fly ash is the ash obtained by the combustion of coal and lignite at thermal power stations. Since they are carried along with the flue gases, electrostatic precipitator is used for collecting the fly ash. The particle size of fly ash ranges between 0.5 µm to 300 µm. Since the heat of hydration is less for PPC, nowadays it is more preferred for constructing mass concrete structures. Because of the microfiller effect and due to the pozzolanic activity of the flyash, the strength and durability of the flyash blended hardened cement matrix will be improved (Nath and sarker, 2011; Wang et al., Part 1, 2003; Wang et al., part II and III, 2004). Before using any super plasticizer with cement its compatibility with that particular cement at given test condition need to be tested (Jayasree 2008, Dhanya et al 2016) because incompatible combination can be resulted in adverse effect like bleeding, retardation in setting, air entrainment etc.

2. Experimental investigation:

2.1 Materials

Locally available Portland pozzolana cement (PPC), River sand of maximum size 4.75 mm , Coarse aggregate of maximum size 20 mm (4.5 mm to 12.5 mm - 80%, 12.5mm to 20 mm – 20%), water, Ligno sulphate based super plasticizer and Black liquor pulp were used for this investigation. Properties of the cement, super plasticizer and aggregates tested according to the codal specification of IS 4031: 2005, IS 9103:2004, IS 2386: 2007. Properties of cement and aggregate obtained from the lab test is checked with the specification given in IS 1489 and IS 383 code respectively. Specification of black liquor pulp provided by the manufactures is given in Table 1.

Table: 1. Properties of black pulp liquor

Tests	Results
p ^H	12.5
Density (kg/m ³)	7.8
Total Solids (%)	9.5
Total Suspended Solids	127
(ppm)	

2.2 Mixing sequence

As the mixing sequence affect the properties of mortar and concrete a uniform mixing sequence was adopted for preparing the mixes. Portland pozzolana cement and aggregate were dry mixed first for two minutes and then 70 % of total mixing water was added to the mix and mixed for 1 minute. Chemical additives (super plasticizer or black liquor pulp) was added to 30% of the mixing water and then poured in to the wet mixed material kept in the mixer and machine mixed for 2 minutes.

2.3 Testing details

Tests performed on cement mortar and concrete are explained in this section.

2.3.1 Tests on Mortar

Portland pozzolana cement mortar of cement to sand proportion 1:1.5 was prepared at a water-cement ratio of 0.4. Maximum amount of black liquor pulp added to the mix was limited to 6% because addition of higher amount of black pulp liquor in concrete leads to decrease in workability. Table: 2 show the mix design of mortar.

Table 2: Mix proportion of mortar

% Replacement of water with black pulp liquor	0%	1%	2%
Cement (kg/m ³)	728.86	728.86	728.86
Black Pulp liquor (ml)	0	1	2
Fine aggregate (kg/m ³)	1093.3	1093.3	1093.3
Water (ml)	100	99	98

Fresh and hardened stage properties of the mortar were prepared by conducting Flow table test and compressive strength tests respectively in lab.

(a) Flow Table Test

Flow table tests on mortar were performed according to the codal provision of IS 1199:2004. Prepared mortar was filled in layers in the conical mould of the flow table of standard dimension (70 mm internal diameter on the top end, 100 mm internal diameter on lower end, and 50mm height). Experimental set up is shown in the figure: 1, after lifting the mould vertically, 25 times jolting was given to the mortar mix. Average spread diameter was measured and flow percentage with respect to the initial diameter of the base of the mould was calculated

b) Compressive strength of Mortar.

Mortar cubes of size 70.6 x 70.6 x 70.6 mm were prepared and tested according to the codal provisions of IS 4031-Part 6 -2005 after giving a 28 day fresh water curing. Compressive strength of mortar was calculated by taking the average of the strength of 3 specimens tested in the lab.

2.3.2. Tests on concrete. A concrete Mix (M30) designed according to the codal provisions of IS10262:2009 is adopted in this investigation. Replacement of water with Black liquor is done till 5%. Table: 3 shows the mix design of concrete with 0, 1, and 2 percentage replacements of water with black pulp liquor.

Table: 3 Mix design for concrete

Dosage	0%	1%	2%
Cement (kg/m^3)	450	450	450
Black Pulp liquor (kg/m^3)	0	1.86	3.72
Fine aggregate (kg/m^3)	619.97	619.97	619.97
Coarse aggregate (kg/m^3)	1182.276	1182.276	1182.276
Water (kg/m^3)	186	184.14	182.28

Following test were performed to determine the fresh and hardened stage properties of concrete.

a) Slump test

Workability of the concrete mixes with 1%, 2%, 3%, 4% and 5% replacement of water with black liquor was determined by conducting slump test according to the codal provisions of IS 1199-2004. Mould of standard dimension (IS 7320-2008) was filled with concrete mix in four equal layers and each layer was tamped properly. Height of subsidence of concrete when mould lifted vertically was measured and reported as slump of concrete.

b) Setting time

Setting time of the concrete was determined by conducting tests according to the codal provisions of IS 8142-2002 using penetration resistance apparatus. Mortar part of the concrete mix was separated from the concrete and filled in the mould. Penetration resistance of the needle to penetrate 2.5 cm in the mortar was measured. Measurement was taken at different time interval and graph was plotted between penetration resistance and elapsed time. Time of initial and final setting of the concrete can be calculated by averaging the elapsed time determined from the curve corresponding to a penetration resistance of 3.43 N/mm^2 and 26.97 N/mm^2 respectively.

c) Compressive strength

Compressive strength of concrete with different replacement levels were determined according to the codal provisions of IS 516-2004. Concrete cubes of 100 x 100 x100 mm size is prepared and tested after 28 day of curing.

d) Split tensile strength

Split tensile strength of the concrete samples were tested according to the codal provisions of IS 5816 -2004. Cylindrical specimen of 150 mm diameter and 300 mm length was

Used for this test. Load required to split the specimen was measured and split tensile strength was calculated.

e) Flexural strength

Flexural strength of the concrete specimen were calculated by conducting flexure test on concrete beam of dimension 150 × 150 × 700 mm according to the codal provisions of IS 516-2004. 3 beams were tested for each mix and average strength was calculated.

2.3.3. Tests on self-compacting concrete. The effectiveness of processed and unprocessed black liquor pulp in preparing self compacting concrete is checked by using raw black liquor pulp and processed black liquor pulp (ligno-sulphate based super-plasticizer) in SCC mix. The SCC mix was prepared by following the guidelines of EFNARC. The ratio of fine aggregate to total aggregate proportions used in the mix was checked with the results of the experiments (Figure:1, Figure: 2) conducted in the lab to determine the optimum percentage of sand proportion to get maximum bulk density and minimum void ratio.

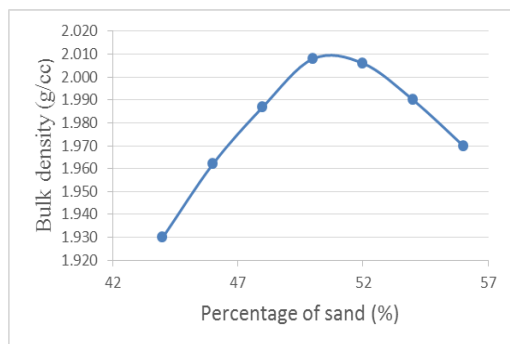


Figure: 1 Bulk density of aggregate with different percentage of fine aggregate

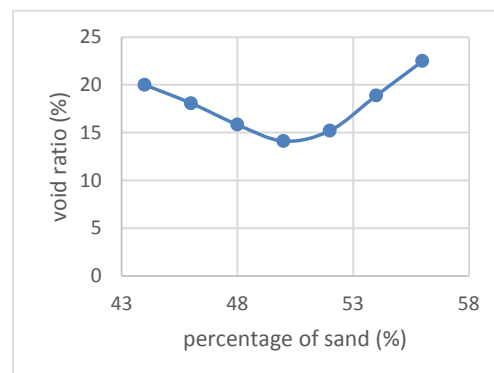


Figure: 2 void ratio of aggregate with different percentage of fine aggregate

From the above graph it is clear that the optimum dosage of sand content for getting maximum bulk density and minimum void content is 50%. That quantity is nearly equal to the adopted value of 53% of sand in the designed mix of SCC. Table: 5 shows the mix design of self compacting concrete using black pulp liquor.

Table: 5. Mix design for self-compacting concrete using black pulp liquor		Table: 6. Mix design for self-compacting concrete using lignosulphate	
Ingredients	Quantity	Ingredients	Quantity
Cement (kg/m ³)	550	Cement (kg/m ³)	550
Fine aggregate (kg/m ³)	860	Fine aggregate (kg/m ³)	860
Coarse aggregate (kg/m ³)	780	Coarse aggregate (kg/m ³)	780

Water (kg/m ³)	220	Water (kg/m ³)	248
Water/cement ratio	0.4	Water/cement ratio	0.45
		Super plasticizer/cement ratio	0.5
		V.M.A/cement	0.2

Black pulp liquor is an unprocessed ligno sulphate based admixture. For comparison, self-compacting concrete is made using commercially available ligno sulphate super plasticizer also. Table: 6 shows the mix design for self-compacting concrete using ligno sulphate based super plasticizer.

Following test were performed to find properties of self-compacting concrete:

- Slump flow test
- J-ring
- L-box
- V-funnel
- Compressive strength

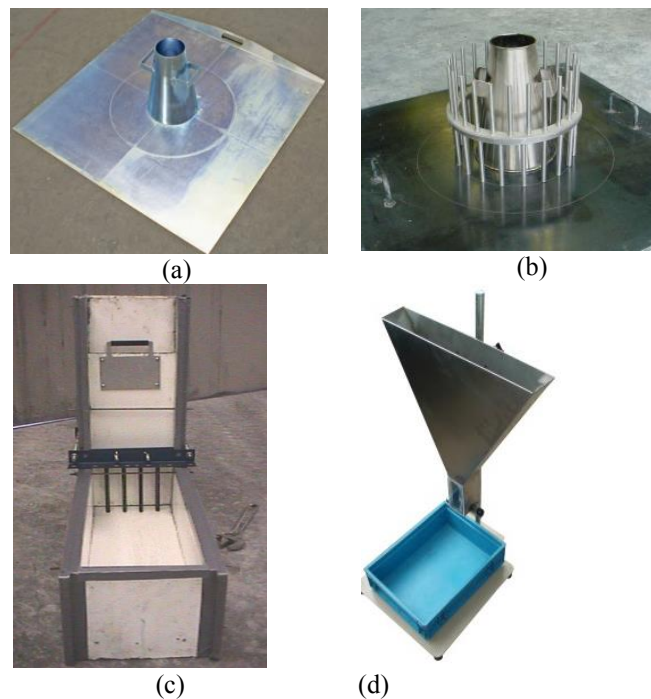


Figure: 3. a) Slump flow test b) J-Ring test c) L-Box test
d) V-Funnel test

Flowability was checked using slump test, the passing ability was determined using J-ring and L-box. The flowability and segregation resistance was determined using V-funnel. Compressive strength of the SCC mix was measured to study the hardened properties of the self-compacting concrete. All the test results were checked with the specification given in UNI 11040.

a) Slump flow test

Slump flow tests were conducted according to the codal provisions of UNI11041 to determine the flowability of self-compacting concrete. Slump flow mould was filled with SCC. Spread diameter and

the time required for the concrete to reach 500 mm diameter when the slump cone is lifted vertically was measured.

b) J-Ring Test

Passing ability of the SCC was tested according to the codal provisions of UNI 11045. The SCC mix is filled in the slump cone kept inside the J-Ring. Cone was lifted vertically and concrete was allowed to flow out through the J-ring. The difference in height between the concrete just inside J-Ring bars and just outside the J Ring bars are measured once the flow is stabilized. The average of the height difference measured in four locations was calculated.

c) L- Box Test

Passing ability of the SCC was tested according to the codal provisions of UNI 11043. Concrete was filled in the vertical deposit of the L-box placed over horizontal surface. After filling the concrete the trap door was opened and allowed the concrete to flow outer. Once the concrete has come to a resting position, the heights of concrete in the vertical portion (H1) and the end of the horizontal portion (H2) of the apparatus was measured. The ratio H2/H1 was used as a measurement of passing ability.

d) V-Funnel Test

Flowing ability and segregation resistance were measured according to the codal provisions of UNI 11042. The SCC mix was filled in the V funnel. The trap door was opened and allowed the concrete to flow out. Time required to empty the funnel was measured. To check the segregation resistance the test was also performed after letting the concrete to rest in the funnel for 5 minutes.

e) Compressive Strength

The 28th day compressive strength of the concrete cube of size 100 x 100 x 100 mm was tested using the provisions of IS 516-2004. Average of the compressive strength of the three specimen tested in the lab was calculated.

3. Results and discussion:

Results of the test conducted in mortar, concrete and SCC are discussed in this section.

3.1 Studies on mortar

Observations of flow table test and compressive strength of the mortar are given in this section

3.1.1 Flow table test on mortar. Flow percent of mortar measured according to the codal provisions of IS 1199:2004 are shown in the figure: 4

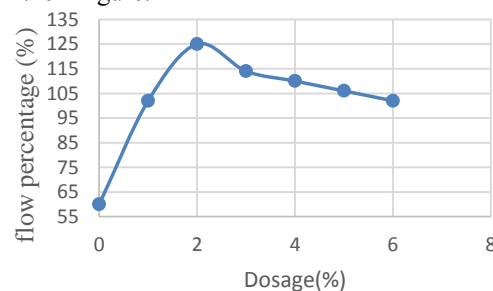


Figure: 4. Flow percent of mortar for different mixes

From the results it was inferred that, replacement of 1% and 2%, there is a steep increase in the workability, though there was a considerable decrease in flow percent after 2% replacement. With 2% replacement, a maximum of 125% flow percent was observed.

3.1.2 Compressive Strength of mortar. Compressive strength of mortar mix 1%, 2%, 3% and 4% replacement of water with black pulp liquor were measured at 7th day and 28th day of curing period. In both case their compressive strength was found to be higher than the control mix at 1%. Maximum compressive strength was observed at 1% replacement.

Figure: 5. shows the compressive result for varying mix of black pulp at 7 and 28 days curing period.

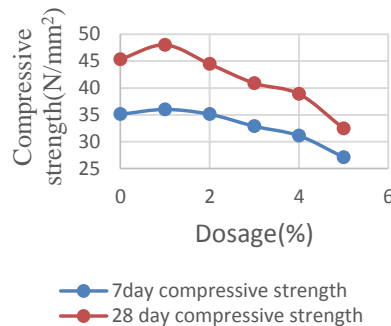


Figure: 5. Compressive strength of mortar

3.2 Studies on concrete

3.2.1 Slump test. Workability of the concrete mixes with 1%, 2%, 3%, 4% and 5% replacement of water with black liquor, determined according to the codal provisions of IS 1199-2004 is shown in the figure: 6. It was observed that there is increase in workability in all replacement levels of black liquor than control. There is consistent reduction in slump for all concrete mixes beyond 2 % replacement of water with black liquor. The maximum slump for concrete mix of 2% replacement of water with black liquor was observed.

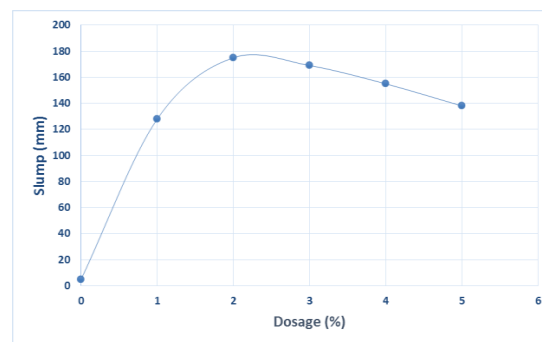


Figure: 6. Slump of concrete

3.2.2 Compression test. Compressive strength of concrete with different replacement levels determined according to the codal provisions of IS 516-2004 is shown in the figure: 7. It was observed that with 1% replacement of water by black pulp liquor, maximum compressive strength was observed. Compressive strength of Concrete mixes 2%, 3%, 4% and 5% was observed to have less strength when compared with the control mix. Figure 4 shows the result of compression test of 7 and 28 days curing period of various concrete mix

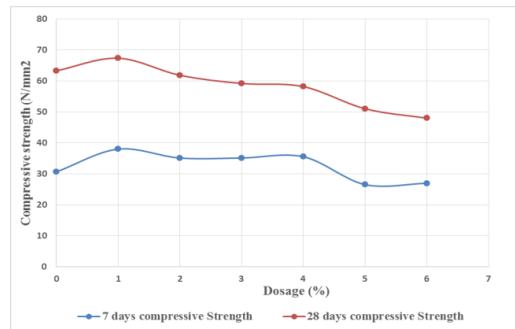


Figure: 7. Compressive strength of concrete

3.2.3 Split tensile strength. Tensile strength of the concrete determined according to the codal provisions of IS 5816 is shown in figure: 8. Maximum split tensile strength was observed for concrete mix with 1% replacement of water with black liquor. Split tensile strength of Concrete mixes with 1% and 2% replacement of water with black pulp liquor was found to have more strength than control mix and beyond 2 % the strength was found to have less than control mix .

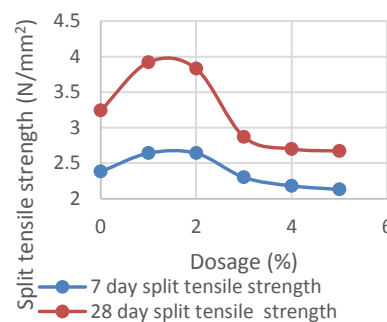


Figure: 8. Split tensile strength of concrete

3.2.4 Flexural strength. Flexural strength of concrete with different replacement levels of black liquor pulp determined according to the codal provisions of IS: 516-2004 is shown in the figure 9. It was observed that maximum flexural strength is observed for concrete with 1% replacement of water with black liquor. Flexural strength of concrete mix 2 % and 3% of replacement of water by black pulp liquor was found to have less strength than control mix

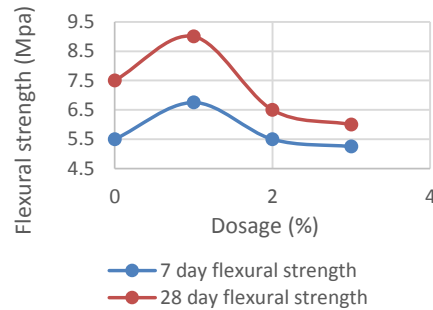


Figure 9. Flexural strength of concrete

3.2.5 Setting time of concrete. Initial and final setting time of concrete determined according to the codal provisions of IS 8142-2002 are shown in the figures 10, 11 and 12 respectively. Figure: 10 shows penetration resistance of mortar at different intervals of time for control mix.

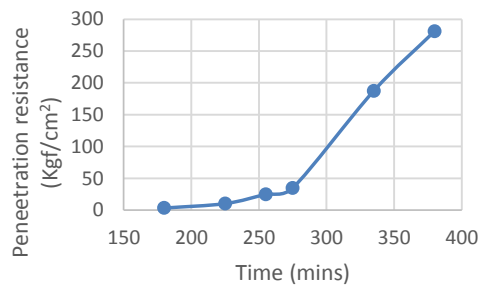


Figure 10 Initial and final setting time of concrete for control mix

From the figure, initial setting time and final setting time of concrete obtained are 280 minute and 365 minute respectively. Figure: 11. Show the penetration resistance of mortar at different intervals of time for 1% replacement of water with black pulp liquor.

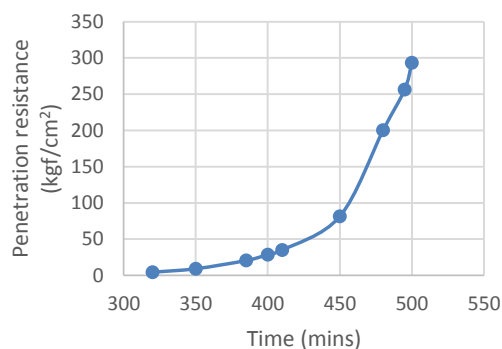


Figure 11. Initial and final setting time of concrete for 1% dosage

From the figure11 initial setting time and final setting time of concrete obtained are 415 minute and 485 minute respectively. Figure: 12. Shows penetration resistance of mortar at different intervals of time for 2% replacement of water with black pulp liquor

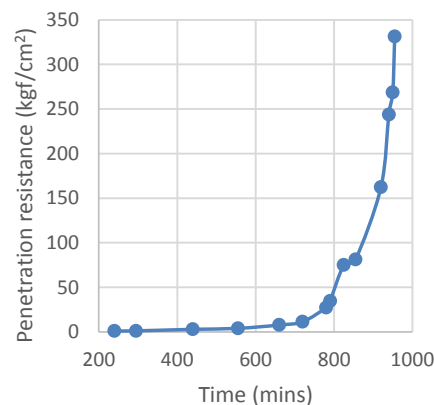


Figure: 12. Initial and final setting time of concrete

For 2% dosage

From the figure 12, initial setting time and final setting time of concrete obtained are 790 minutes and 952 minutes respectively.

Increase in the replacement of water with black pulp liquor increases setting time of the concrete. With respect to control mix the initial setting time of 1% replacement of water with black pulp liquor was delayed by 135 minutes and final setting time was delayed by 120 minutes. With respect to control mix the initial setting time of 2% replacement of water with black pulp liquor was delayed by 510 minutes and final setting time was delayed by 587 minutes. Maximum setting time was observed for 2% replacement of water with black pulp liquor.

3.3 Studies on self compacting concrete

Tests were conducted on self-compacting concrete prepared at a water cement ratio 0.4 using processed and unprocessed black liquor pulp and it was found that the mixes were not satisfying the acceptance requirement of SCC. To check the effectiveness of the processed and unprocessed black liquor pulp in high performance concrete, the SCC were prepared at higher water cement ratio of 0.45 and it was found that processed admixtures are good enough in preparing SCC of desired property. But unprocessed super-plasticizers are not good in preparing SCC mix even at higher water cement ratio. Measured Properties of the SCC prepared using processed black liquor pulp are given in this section.

3.3.1 Slump flow test. Result of Slump flow tests shows that the time corresponding to 50 cm spread and final spread diameter are 1.5 sec and 67 cm respectively.

The acceptable range of slump flow spread diameter is 65 – 80 cm. These results are within the acceptable limit.

3.3.2 J-Ring Test. Passing ability of the SCC tested according to the codal provisions of UNI 11045. Difference in height of concrete just inside and outside the J ring is found to be 0.78 cm. The results of J-ring test shows that, passing ability of concrete were in the acceptable range (0 – 10 mm).

3.3.3 L-Box test. Passing ability of the SCC was tested according to the codal provisions of UNI 11043. The ratio of height of concrete at the end of horizontal section (H2) to the height of concrete at

the vertical portion of the L – Box is found to be 0.68 which shows that the passing ability of concrete was not in the range (0.8 – 1.0).

3.3.4 V-Funnel Test .The results obtained from V-funnel test shows that the time taken to empty the concrete in the V funnel at the beginning and after 5 sec is found to be 3.4 sec and 5.59 sec respectively. Hence, difference between times is 2.19 seconds, which shows segregation resistance of the concrete is in acceptable range (3 - 5).

3.3.5 Compressive strength .The 7th and 28th day compressive strength of the concrete cube tested according to the provisions of IS 516 are found to be 29 Mpa and 37.5 Mpa respectively.

4. Conclusion

From the experimental investigation on cement mortar it was found that, maximum workability was observed for 2% replacement of water with black pulp liquor. For 1% replacement of water with black liquor, maximum compressive strength was observed. From the study on concrete it was found that, maximum workability was observed for 2% replacement of water with black pulp liquor. For 1% replacement of water with black liquor, maximum compressive strength, flexural strength and split tensile strength was observed. Initial and final setting times were delayed with addition of black pulp liquor. For 2% replacement of water with black liquor, the maximum setting time was observed. Even with 20% addition of black liquor, satisfactory flow for self-compacting concrete was not observed. Hence we can conclude that 1% replacement of water with black pulp liquor increases the fresh properties of the concrete, 2% replacement of water with black pulp liquor increases the mechanical properties of the concrete and acts as a set retarder. Black pulp liquor has lower performance compared to the commercially available lingo sulphate admixture for making self-compacting concrete and it is observed that unprocessed black liquor pulp is not effective in producing self compacting concrete.

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