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Production and Utilization of Retarder from Hardwood Sawdust

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ABSTRACT

In this study, production of retarding admixture (ASTM C494 Type-B) from hardwood sawdust and utilization on concrete by using various dosage of admixture was studied. In production of retarder as a local product lignosulphonates based retarding admixture was produced to investigate differences of test results by using admixtures. In this project, selected raw material was Pyin-Ka-do sawdust and sodium sulphite (Na_2SO_3) was used as a cooking aid chemical liquor by heating at 110°C based on pulp and paper sulphite process. The mechanisms of action of lignosulphonate admixture in cement-water systems are prescribed by five different types interaction between retarder and cement grains : (i) Reduction in surface tension of water (ii) Adsorption (iii) Electrical repulsion (iv)Dispersion and (v) Deflocculation. These reactions cause delaying in setting time.

In utilization of retarder on concrete, setting time of hydraulic cement paste, compressive strength of mortar and concrete were considered by using produced admixture dosage of (0.5%, 1%, 1.5%) by weight of cement. The setting time testing of cement paste was made by using vicat needle penetration test, and compressive strength was tested with standard cubes. The test results of produced admixture were compared with ASTM C494 (1993). Compressive strength of mortar was tested with 3 inches cube and that of concrete was tested by 6 inches standard cube. For comparison of cost and results of produced admixture, commercial admixture (0.5% by weight of cement) was tested.

KEYWORDS: *compressive strength, setting time, lignosulphonate, retarder, admixture*

I. INTRODUCTION

In Myanmar, chemical admixtures are widely used for obtaining the desirable results of the job site requirements. The specific quality of concrete depends on materials, mix design, production, transporting, placing and curing. During mixing of concrete, cement reacts with water and occurs exothermic hydration reaction. The addition of admixture to the concrete affects the hydration process of the cement. In the fresh stage of mixing, the mix should be workable enough to be compacted. In the hardened stage, satisfactory compressive strength is needed. Thus the use of admixture should not adversely affects to the properties of concrete.

In Myanmar, the organic chemical admixtures are imported from other countries and they cannot be produced as a local product. As a result, it causes to loss of foreign currency and higher cost for applicators. Cement- admixture may occur incompatible problem by using with local cement. This is due to the specific effect of all types of admixtures are mainly dependent on composition of cement. In this research, lignosulphate, organic based retarding admixture was produced and utilized on concrete by using locally available cement.

II. LITERATURE REVIEW

A. General

An admixture can be defined as any materials other than water, aggregate and hydraulic cement which is used as an ingredient of a concrete or mortar mixture and is added to the batch immediately before or during its mixing. Admixture may be used to modify the properties of concrete at fresh and hardened stage. The useful classes of admixture for concrete are organic chemical materials and active mineral powders such as pozzolan, Admixtures are available as both liquid and powders. Water-soluble powders should be dissolved in water prior to use so that they can be dispensed as liquids. Powder admixtures that are not completely soluble in water should be added to the fine aggregate. In the application of liquid type admixtures, it is important to avoid adding them directly to the dry and absorptive aggregates.

B. Hydration of Cement

Admixture that modifies the properties of fresh concrete may cause problems through early stiffening or undesirable prolongation of setting times. The cause of abnormal setting behaviour should be determined through studies of how such admixtures affect the hydration of cement.

All main phases of Portland cement [tricalcium aluminate (C_3A), tricalcium silicate (C_3S), dicalcium silicate (C_2S) and tetracalcium alumino ferrite (C_4AF)] evolve heat when they react with water [and calcium sulphate (gypsum)] and this chemical reaction which leads to hardening and development of strength is called hydration and the heat generated during this reaction is called heat of hydration.

1. Aluminate Phase

Immediate reaction with water and calcium sulphate (gypsum) forms a compound called ettringite [$3CaO.Al_2O_3.3CaSO_4.32\ H_2O$]. In the absence of calcium sulphate, C_3A reacts directly with water to form $3CaO.Al_2O_3.6H_2O$ (C_3AH_6). This is a very rapid and exothermic reaction that leads to a quick hardening known as 'flash set'. The ettringite coating the underlying C_3A then reacts with it and additional water to form a low sulphate ettringite. The approximate reaction being

$$C_3A + 6H \rightarrow C_3AH_6$$
$$[100]\ [40]\quad [140]$$

2. Silicate Phase

The product of hydration of C_3S is the microcrystalline hydrate with some lime separating out as crystalline Ca (OH_2) [calcium hydroxide], C_2S behaves similarly but clearly contains less lime. The calcium silicate hydrate, 3CaO.2SiO2. $3H_2O$ is described as C-S-H (tobermorite gel). The approximate hydration reaction being written as follows:

$$\text{For } C_3S;\ 2C_3S + 6H \rightarrow C_3S_2H_3 + 3Ca(OH)_2$$
$$[100]\ [24]\quad [75]\quad [49]$$

$$\text{For } C_2S;\ 2C_2S + 4H \rightarrow C_3S_2H_3 + CaOH_2$$
$$[100]\ [21]\quad [99]\quad [22]$$

The numbers in the square brackets are the corresponding masses.

C. Active Ingredients used in Retarding Admixture

The materials those are generally available for use in retarding admixtures can be divided into five groups.

1. Lignosulphonic acid and their salts
2. Modification and derivatives of the first group
3. Hydroxylated carboxylic acids and their salts
4. Modification and derivatives of the above group
5. Other material which include (i) inorganic materials (ii) Amines and their derivatives (iii) Carbohydrates, polysaccharides and sugar acids (iv)certain polymeric compounds.

In this research, produced lignosulphate admixture is organic admixture.

D. Use of Retarder in Concrete

The desirable property of concrete in the fresh state is that it should have adequate fluidity without segregation and bleeding, place ability, compatibility and finish-ability appropriate to the conditions of placement. These properties are collectively called (workability).

Retarding water-reducing admixtures are used to improve the quality of concrete, to obtain specified strength at lower cement content and to increase the slump of a given mixture without increasing water content and delay in setting. So, they may be used to offset the accelerating effect of high ambient temperature, to eliminate the formation of cold joints and discontinuities, to transport concrete over large distances and so on.

E. Mechanisms of Action for Lignosulphonate Based Retarder in Hydration of Cement

The surface-active properties of lignosulphonates in cement-water system are as follow.

A. Reduction in surface tension of water
B. Adsorption on C_3A and C_3S phase of cement
C. Electrical Repulsion (large negative zeta-potential)
D. Dispersion
E. Deflocculation

The surface activity of lignosulphonates is due to an unbalanced charge of electricity carried by these. Due to their ability to migrate to the water-air boundary, plasticizing water-reducers reduce the surface tension at the boundary. Such lowering of surface tension aids better wetting and hydration reaction.

In cement water dispersion, the tail end of the surfactant gets adsorbed onto the cement particles. These fine particles of cement in water would tend to floc together because of physical and chemical forces of attraction between them would be dispersed. At the same time, the water trapped inside such a floc of cement particles gets released.

Due to adsorption of lignosulphonate ions on them, the cement particle develops similar charges and repels each other. A film is formed around the cement grains which prevents or reduces the hydration reactions with water.

F. Behavior of Materials used in Concrete

1. Cement

The various types of cement have the property that when mixed with water, a chemical reaction (hydration) takes place and produces a very strong binding medium for the aggregate particles.

The physical properties of Portland cement are fineness, setting time, consistency, specific gravity, compressive strength.

A. Fineness Test

Fineness is expressed in percent retaining on a sieve of ASTM No.200 or B.S. No. 170 after shaking for a period of 15 minutes. It shall not exceed 10%. In this study, fineness of Myinegalay cement (Type I) is 8%.

B. Consistency Test

Consistency test is to determine the percentage of water required for preparing cement paste of standard consistency is between 26 and 33%. Standard consistency of Myinegalay cement is 32%.

C. Setting Time Test

Setting is caused by hydration of cement compounds (C_3A and C_3S). ASTM prescribes C191-92 the minimum initial setting time of 60 minutes and final setting time as a maximum of 10 hrs for Portland cement. In this test, initial setting time and final setting time are 1:37 and 2:04 (hr:min).

D. Compressive Strength Test

Two British standard methods for testing compressive strength of cement are mortar testing and concrete testing. Compressive strength of Ordinary cement for (1:3) mortar mix at 3-days and 7-days curing should not be less than 1200 psi and 2000 psi [reference in ASTM (1975)].

TABLE I. Compressive Strength of Cement

Sample No	3-days (psi)	7-days (psi)
1.	1421	1653
2.	1465	1710
3.	1450	1697
4.	1446	1784
Average	1445	1711

2. Aggregate

Aggregates are generally classified as fine and coarse. Fine aggregate or sand is any material that passes a No.4 ASTM sieve and retain on No.200 sieve. Material coarser than this is classified as coarse aggregate or gravel.

The physical properties of aggregates are specific gravity, sieve analysis, absorption and moisture content.

Specific gravity is defined as the ratio of mass of a unit volume of material to the mass of the same volume of water at the stated temperature. The majority of natural aggregates have an apparent specific gravity between 2.6 and 2.7.

The process of dividing a sample of aggregate into fractions of same particle size is known as sieve analysis. It is made for the purpose of fineness modulus and its typical values range from 2 to 3.5 for fine aggregate and for coarse aggregate from 5.5 to 8.

The water absorption is determined by measuring the decrease in mass of saturated and surface dry sample after oven-drying for 24 hours. The ratio of the decrease in mass to the mass of dry sample, expressed as percentage, is termed absorption. Moisture content is defined as the water in excess of saturated and surface dry condition.

TABLE.II Test Results for Ordinary Portland Cement (Rhinoceros Brand)

Specific Gravity	Fineness	Standard Consistency	Setting Time (hr:min)		Compressive Strength (psi)	
			I.S	F.S	3-days	7-days
3.08	*	32	1:37	2:04	1445	1711

Remark:

I.S = Initial Setting Time
F.S = Final Setting Time
* 92% passing BS sieve No.170

TABLE III. Test Results for Fine and Coarse Aggregates

Sample Name	Fineness Modulus	Specific Gravity	Density (g/cc)	Absorption	Moisture Content
Yangon River Sand	2.65	2.5	2.5	2.05	5.13
Yangon River Shingle	7.5	2.63	2.63	0.715	Nil

III. Production of Retarding Admixture

A. Reaction of Lignin in Sulphite Process

Lignin is the most abundant constituent of the woody structure of higher plants. It comprises 20 to 40% of the mass of dry wood. Lignin acts as a cementing medium to bind the matrix of cellulose fibres together in a rigid woody structure. Lignin content of the plants is as follows: (i) softwoods (27~30%), (ii) hardwoods (16~24%), (iii) bamboo, other grasses, cotton stalk, wheat and rice straw (11~20%).

The successive steps in degree of acidity in sulphite cooking liquors, using sodium as the base, have been designated as follows:

A. Acid-bisulphite ($NaHSO_3 + SO_2$)
B. Bisulphite ($NaHSO_3$)
C. Bisulphite- sulphite ($NaHSO_3 + Na_2SO_3$)
D. Sulphite (Na_2SO_3)
E. Neutral sulphite ($Na_2SO_3 + Na_2CO_3$)

The primary reaction of the sulphite process occurs between the sulphite ions of the cooking liquor and the lignin of the wood. The reaction forms lignin sulphonic acids. The rate of sulphonation reaction is dependent on the temperature and the sulphite ion concentration.

In the lignin sulphonation reaction with sulphite cooking liquor, the time consumedin the penetration period of a sulphite cook, which may be as long three hours at 110°C has for economic reasons. The chemical reaction of lignin sulphonation reaction with sulphite cooking liquor can be written as

$HSO_3^- + \text{lignin-OH} \rightarrow \text{lignin-}SO_3^- + H_2O$

B. Production of Sodium Lignosulphonate based Set Retarding Admixture

In this study, wood and chemical reagent are used as raw materials to produce lignosulphonate based on sulphite process. Hardwood (Pyin-Ka-Do) sawdust and sodium sulphite (Na_2SO_3) powder which is used as cooking aid chemical are selected as raw materials.

Quantity of applied materials	: sawdust 454 gm Na_2SO_3 300gm
Temperature	: 110°C
Duration of cooking	: 3hours

For the production of Lignosulphonate, step-by-step procedure is as follows:

1. Cooking liquor is prepared by mixing 300gm of sodium sulphite and 2 liter of ordinary tap water in the steel pot. pH value of fresh liquor is 10. Then 454gm of sawdust which is removed from undesirable materials is added to the cooking liquor. And then the pot is marked at the point where the mixture reached.
2. Mixture of sawdust and cooking liquor is heated by hot plate joining with voltage regulator. The duration of cooking is as long as 3 hours at 110° C .At the early stage of cooking, sulphur dioxide gas is released. It has an undesirable pungent smell.
3. The boiling water is added to cover the loss of water from the cooking liquor due to evaporation. It is added to reach the marking point of the pot whenever the water loses. At the end of cooking stage, cooking liquor emits sweet smell and it has dark brown colour.
4. After that dark brown colour liquid is separated from the solid portion by filtration and squeezing.
5. In this step, liquid lignosulphonate is heated to form dry powder lignosulphonate by evaporation and drying. Dry powder form of lignosulphonate is important for constant solid concentration of liquid admixture and storage life. In this study, liquid type admixture is prepared by adding produced dry powder lignosulphonate with water to get the constant solid concentration of 23 gm of powder per 100 ml of liquid.

C. Quality of Product

Colour : dark brown
Solid Content : 23 gm of powder per 100 ml of liquid
Density : 1.18 kg/li
pH : 10

D. Unit Price of Produced Admixture

Sodium sulphite : 462 ks for 300gm
Sawdust : 10 ks for 454 gm
Temperature : 160 ks for 6.4kwh
Total Cost for an Experiment : 632ks
Yield : 49% powder form (based on sawdust) (approximately)
:820ml liquid type (approximately)

Unit price of produced admixture is 770 kyats per liter.

IV. Effect of Produced Admixture on the Initial Setting Time and Compressive Strength

TABLE.IV Initial Setting Time of Neat Cement Paste with and without Retarding Admixture

Mix	Admixture Dosage %by Weight of Cement	Number of Sample	Average Initial Set (min)	Estimated Final Set (min)
Control	-	10	94	203
Produced Admixture	0.5	10	134	250
Produced Admixture	1	9	162	284
Produced Admixture	1.5	5	194	323
Commercial Admixture	0.5	4	137	254

Remark: Final setting time (min) = 91+1.2 [Initial setting time (min)]

TABLE.V Average Compressive Strength of (1:3) Mortar Specimens

Mix	Admixture Dosage % by Weight of Cement	Water-cement Ratio	No. of Cube Specimen	Average Compressive Strength (psi)		
				3-days	7-days	28-days
Control	-	0.5	12	1445	1711	2137
Produced Admixture	0.5	0.5	12	1457	1769	2130
Produced Admixture	1	0.5	12	1350	2133	2900
Produced Admixture	1.5	0.5	12	1300	1850	2100
Commercial Admixture	0.5	0.5	12	1606	2045	2500

TABLE.VI Mix Proportions and Slump Value of Concrete Mixes

Mix	Admixture Dosage % by Weight of Cement	Mix proportion for a cubic meter (kg)				Effective w/c Ratio	Slump (in)
		Cement	Aggregate		Water		
			Fine	Coarse			
Control	-	308	569	1357	169	0.6	2
Produced Admixture	0.5	308	569	1357	169	0.6	2 ¼
Produced Admixture	1	308	569	1357	169	0.6	2
Produced Admixture	1.5	308	569	1357	169	0.6	2 ½
Commercial Admixture	0.5	308	569	1357	169	0.6	2 ½

TABLE.VII Test Results for Average Compressive Strength of Concrete Specimens

Mix	Admixture Dosage, % by weight of cement	Compressive Strength of Standard Cubes		
		3-days	7-days	28-days
Control	-	1867 1929 1878	2177 2163 2239	2823 2823 2853
	Average	1891	2193	2833
Produced Admixture	0.5	2488 2624 2503	2860 2653 2868	3364 3509 3197
	Average	2538	2794	3357
Produced Admixture	1.0	2892 2923 2933	3102 3316 3194	3129 3283 3168
	Average	2916	3204	3193
Produced Admixture	1.5	1181 1193 1185	1706 1911 1728	3379 3201 3568
	Average	1186	1782	3383
Commercial Admixture	0.5	2425 2498 2488	2742 2965 2742	4166 3940 4203
	Average	2470	2816	4103

V. Comparison of Results

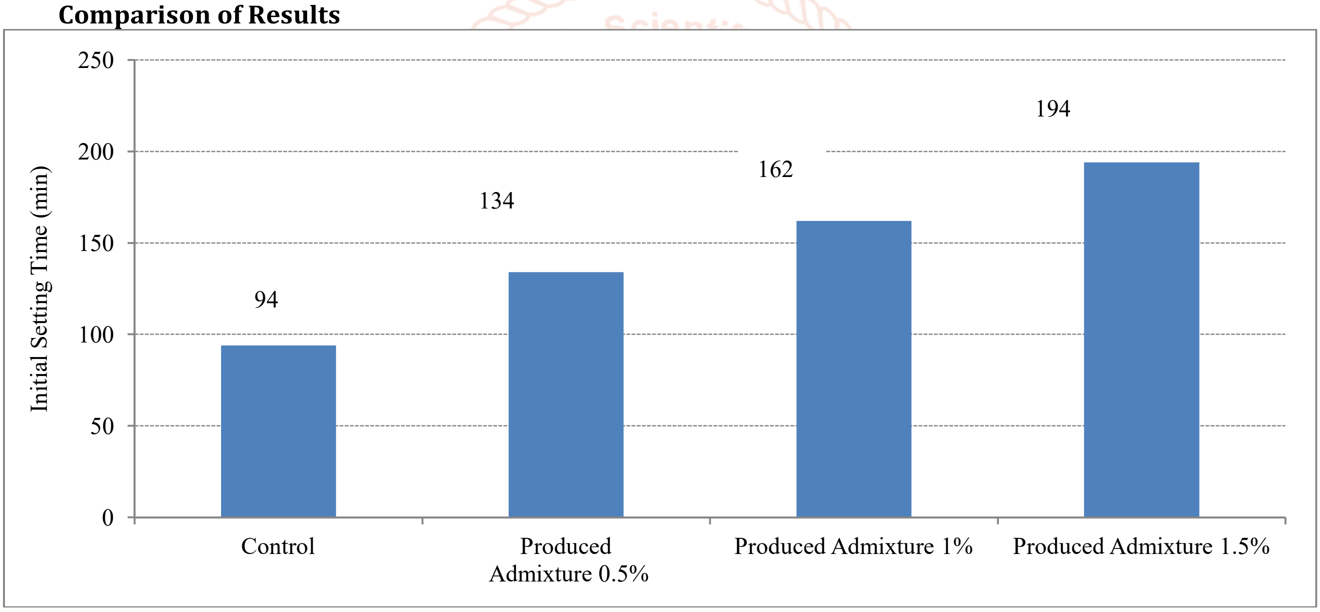

Fig.1. Comparison of Initial Setting Time of Cement Paste with and without Produced Admixture

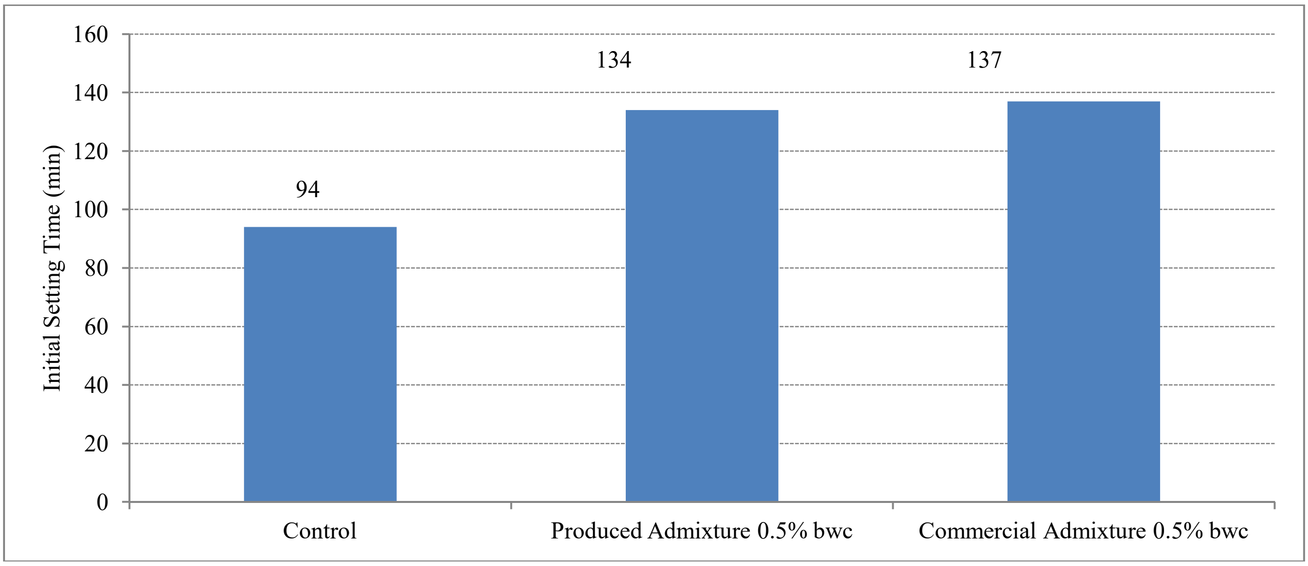

Fig.2. Comparison of Initial Setting Time of Cement Paste with Different Types of Admixture

TABLE.VIII Comparison of Test Results with the Standard Requirement for ASTM C494 Type B and D Admixtures

Mix	Admixture Dosage %bwc	Actual Initial Set Deviation from Control Mix	ASTM C494 Type (B) and (D) Initial Setting Time Allowable Deviation from Control
Produced Admixture	0.5	40 minutes later	At least 1:00 later not more than 3:30 later
Produced Admixture	1	1:08 hour later	
Produced Admixture	1.5	1:40 hour later	

TABLE.IX Comparison of Test Results with Standard Requirement for ASTM C494 Type (A) Admixtures

Mix	Admixture Dosage %bwc	Actual Initial Set Deviation from Control Mix	ASTM C494 Type (A) Initial Setting Time Allowable Deviation from Control
Produced Admixture	0.5	40 minutes later	Not more than 1:00 earlier Nor 1:30 later
Produced Admixture	1	1:08 hour later	
Produced Admixture	1.5	1:40 hour later	

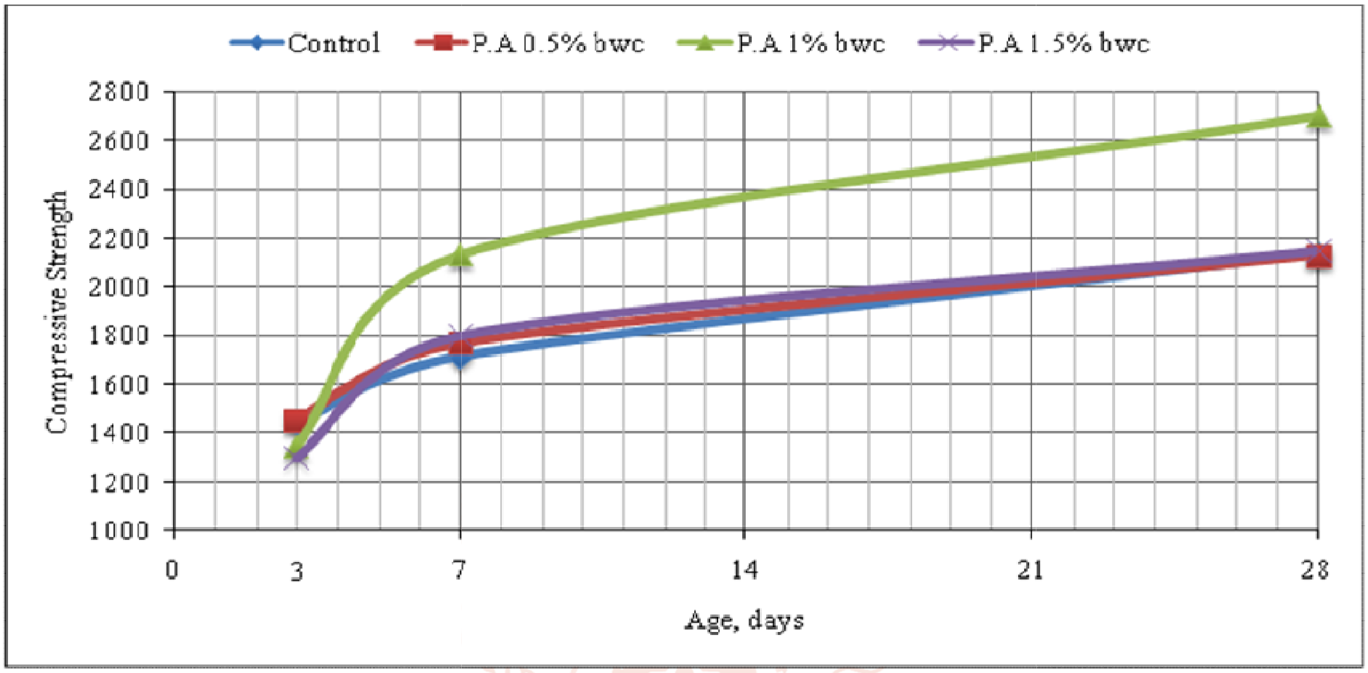

Fig.3. Comparison of Compressive Strength of Mortar with and without Produced Admixture

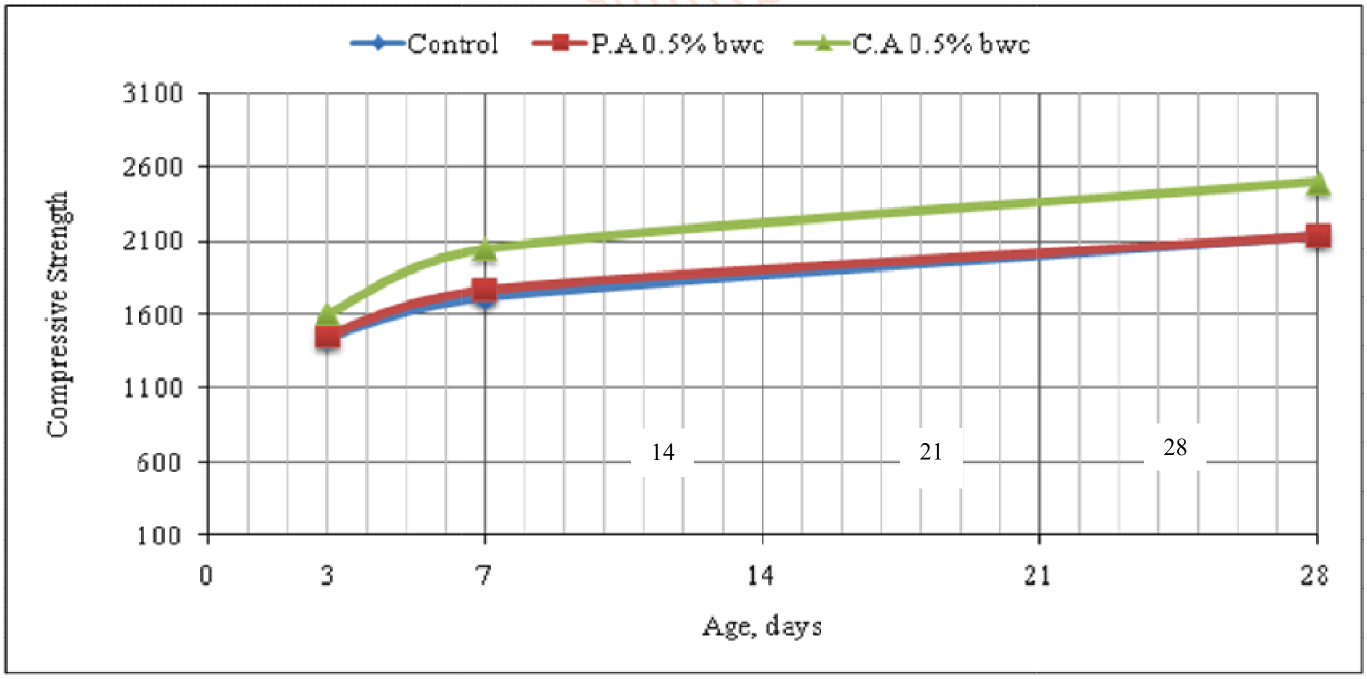

Fig.4. Comparison of Compressive Strength of Mortar with Different Types of Admixture

TABLE.X Comparison of Relative Compressive Strength of Concretes with the Standard Requirement for ASTM C 494 Type (A and D) Admixture

Mix	Admixture Dosage % bwc	Actual Relative Compressive Strength, (%) of Control			ASTM Type (A and D) Minimum % of Control		
		3-days	7-days	28-days	3-days	7-days	28-days
Produced Admixture	0.5	134.2	127.4	118.5	110	110	110
Produced Admixture	1	154.2	146.1	112.7			
Produced Admixture	1.5	62.7	81.2	119.4			
Commercial Admixture	0.5	130.6	128.4	142.32			

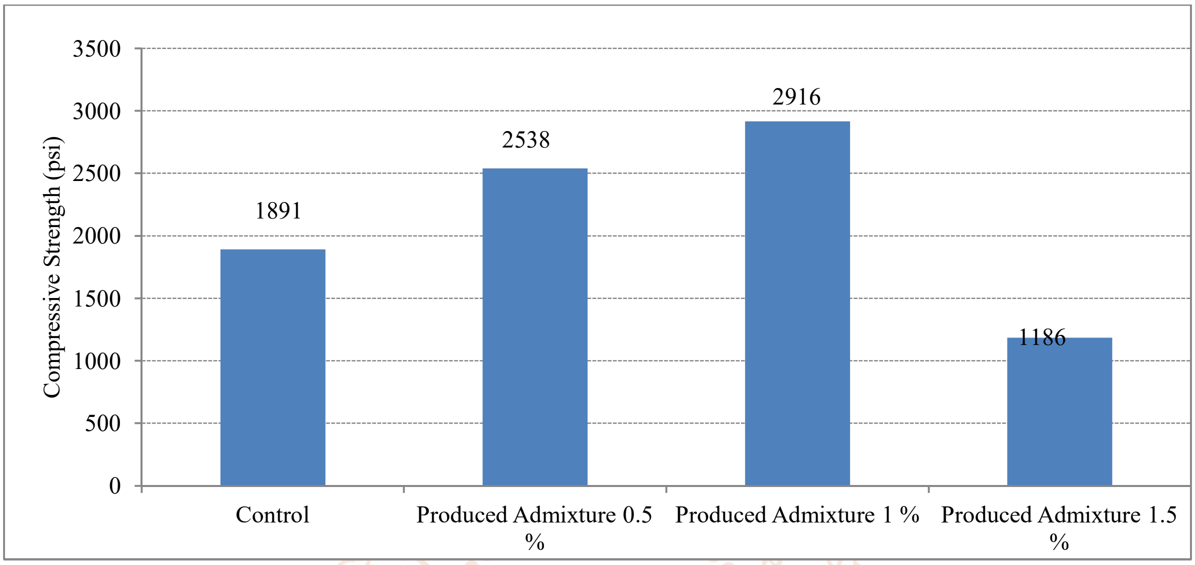

Fig.5. Comparison of Compressive Strength of Concrete with and without Produced Admixture for 3-days

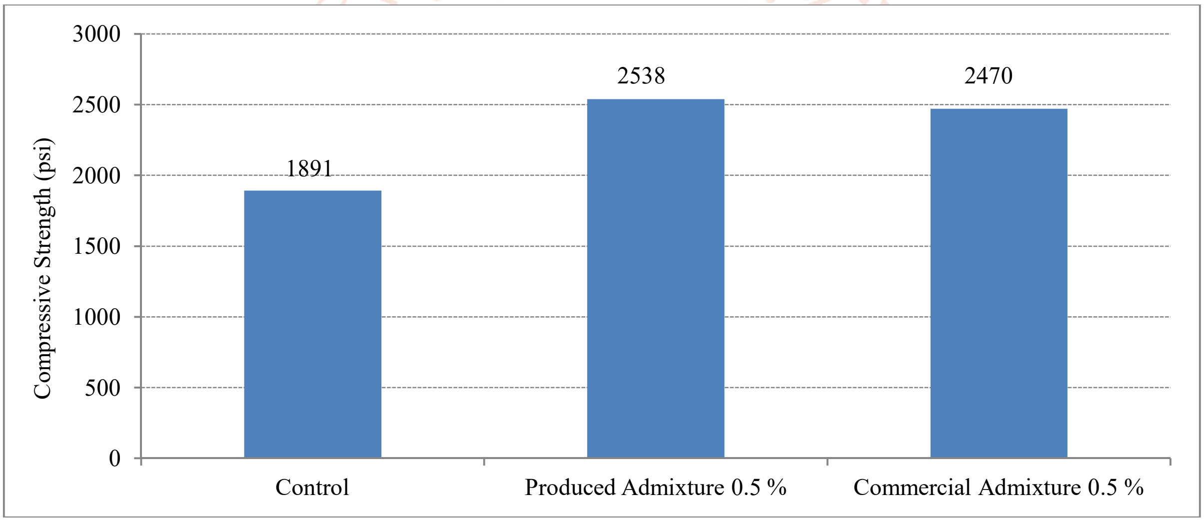

Fig.6. Comparison of Compressive Strength of Concrete with Different Types of Admixture for 3-days

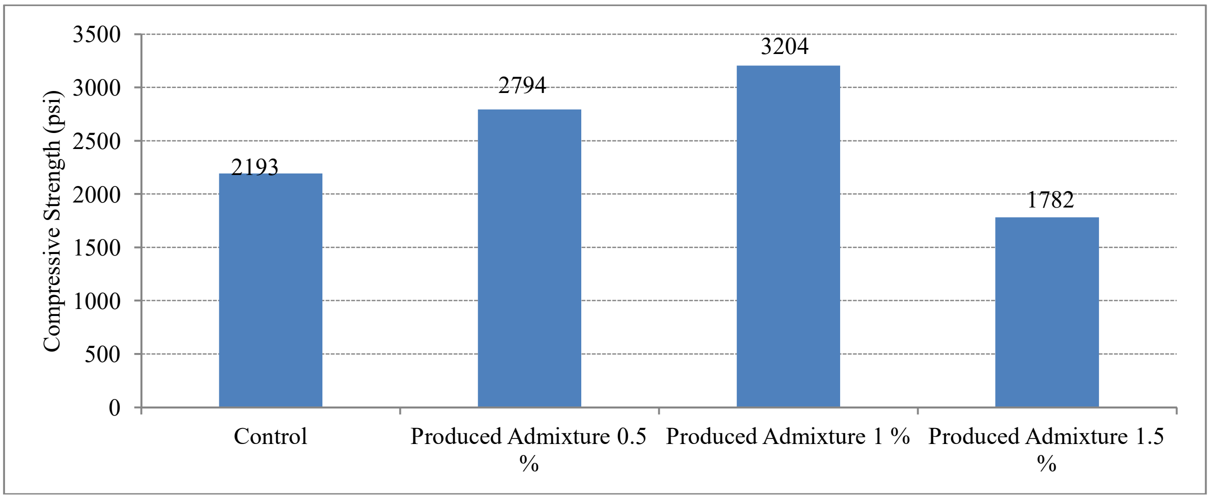

Fig.7. Comparison of Compressive Strength of Concrete with and without Produced Admixture for 7-days

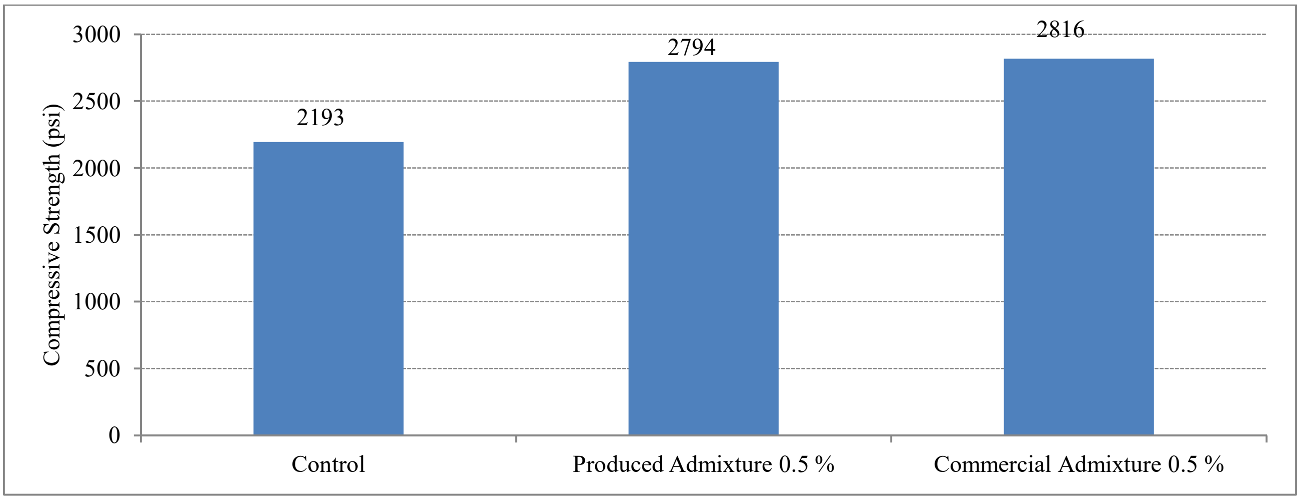

Fig.8. Comparison of Compressive Strength of Concrete with Different Types of Admixture for 7-days

TABLE.XI Comparison of Cost for Admixtures

Particular	Type, according to ASTM C 494	Unit Price
Produced Admixture	Type D	770 kyats per liter
Commercial Admixture	Type B and D	1000 kyats per liter

VI. DISSCUSSIONS AND Conclusions

The efficiency of produced admixture is considerably varied with the composition of raw materials (wood and cooking aid chemical). The highest product efficiency and product yield greatly depend on the rate of reaction of lignin sulphonation. The degree of lignin sulphonation may be high by using high acidity of cooking liquor and the wood containing highest lignin content.

In this study, hardwood sawdust (Pyin-Ka-Do) and sodium sulphite (Na_2SO_3) are used in the production of lignosulphonate. In this case, the variation of physical and chemical properties of lignosulphonate may be less because of lower lignin content of wood and low acidity of cooking aid chemical.

The unit price of produced admixture is 25% less than that of commercial admixture. For more economical and commercial use, sulphurous acid and bisulphite should be used as cooking liquor because of unnecessary of high temperature, long duration time of cooking and improving the rate of dissolving lignin components which improve the quantity of product.

From the discussions in this paper, the following conclusions can be drawn:

1. The production procedure by using hardwood (Pyin-Ka-Do) sawdust and sodium sulphite (Na_2SO_3) as a cooking aid chemical liquor can be successfully used for the production of lignosulphonate based set-retarding and water-reducing admixture.
2. Produced admixture can be effectively used as Type A, water-reducing admixture or Type D, water-reducing and retarding admixture depend on the amount of dosage.
   3. Produced admixture dosage 0.5% and 1% by weight of cement can be used as Type A and Type D. Produced admixture dosage 1% gives the highest compressive strength on concrete according to 3 and 7 days strength. Produced admixture dosage 1.5% cannot be used as admixture because it gives very small amount of compressive strength in spite of delaying in setting time.
4. In making mortar mixes and testing initial setting time, produced admixture can be beneficially utilized.
5. In order to produce more economically and commercially, it should be used pressure digester and high acidity of cooking aid chemical in the production product.
6. To obtain uniform lignin content of wood, the same source of wood is important to be used.

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