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The Effects of Pine (Pinus Canariensis) Tree Bark Extract on the Properties of Fresh and Hardened Concrete

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Abstract
In this research, pine tree bark extract was prepared in a digester by using water, heat, and pressure. In the digester, the ratio of the mass of the bark to that of the water was 1:2, respectively. The temperature in the digester was raised from the ambient level to 121 ^{o} C in 15 minutes. At the temperature of 121^o C, the pressure inside the digester was 0.1 N/mm^2. These conditions of heat and pressure were maintained for a period one hour, resulting in the liquid phase of the mixture turning opaque brown (black liquor). Without further processing, the black liquor was used as a concrete admixture and tests carried out to determine its effects on workability of fresh concrete and strength of hardened concrete. Dosages of the black liquor used, expressed as a mass percentage of the cement content were 10 %, 20 %, 30 %, 40 %, and 47.5 %. In each case, the mixing water was reduced by an amount equal to the mass of the dosage. For each dosage, compressive strength tests were carried out at the ages of 3,7,28, and 61 days. Compared with control concrete, increases of workability were observed for all the dosages. However, consistent improvement in compressive strength only occurred when the dosage of pine bark extract was 20 %.
Keywords: Concrete admixture, lignin, pine, strength, workability.

1. Introduction

Concrete is the most widely used construction material. Depending on performance requirements, it can be produced to different specifications by varying the ingredients and their proportions. Among the important properties of concrete are strength and workability. In this study an attempt is successfully made to develop a technique that can improve these two properties cost effectively, sustainably, and in an eco- friendly manner. To achieve this objective, the effects of the pine tree bark extract, when used as an admixture, on the workability of the fresh concrete, and the strength of the hardened concrete were studied. The pine trees are grown in polar, temperate, and tropical climates of the world. They are widely planted in Kenya for timber production. In the research, a normal concrete mix was designed and prepared as a control. For a test mix, the materials design output of the control was used, with a fraction of the mixing water substituted with an equal mass of the pine bark extract. Five test mixes were made, each containing a different mass of the pine bark extract. Calculated as percentage of the cement content, these masses were equal to 10, 20, 30, 40, and 47.5 percent of the cement content. At the test mix preparation stage, part of the mixing water, at least equal in mass to the amount required to bring the aggregates to saturated surface dry condition was mixed with the weighed aggregates and allowed 5 minutes to soak prior to addition of the cement, pine extract, and the more water necessary for the concrete to attain the target workability of 45 mm slump. The mass of the mixing water less the exact amount required for the absorption was 47.5 % of the mass of the cement content. Hence, at a dosage of 47.5 %, lubrication and hydration water had been completely substituted with pine extract. All the concrete operations were carried out in accordance with the relevant British standards. Strength tests were done at the ages of 3, 7, 28, and 61 days. Slump tests were used to assess the workability of the concrete.

Concrete is made by mixing together, in the right proportions, cement, water, aggregates, admixtures, and additives. For a normal concrete mix, Portland cement is used and neither an admixture nor an additive is added. The two most common types of Portland cements are ordinary Portland cement and rapid – hardening Portland cement. Portland cement is made by burning in a kiln, at about 1450^o C, a mixture of lime (CaO), silica (SiO_2), alumina (Al_2O_3), and iron oxide (Fe_2O_3) to form clinker. Typical oxide proportions are lime (60 to 65 %), silica (18 to 25 %), alumina (3 to 8 %), and iron oxide (0.5 to 5 %) (Kong, F.K., Evans, R.H. 1985, **p. 18**).The different types of Portland cement are obtained by varying the relative proportions of these four oxide compounds and by grinding the clinker to different degrees of fineness. The current Building Research Establishment (BRE) method of normal concrete mix design provides strength data for Portland cement classes 42.5 and 52.5 only. Portland cement strength class 42.5 was used.

The main constituent compounds of Portland cement are tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium aluminoferrite. In the concrete mix, chemical reactions between water and tricalcium silicate and dicalcium silicate take place as illustrated by equations (1.1), and (1.2). Hydration of tricalcium silicate ($3CaO.SiO_2$):

$$2(3CaO.SiO_2) \quad + \quad 6H_2O \quad \rightarrow \quad 3CaO.2SiO_2.3H_2O \quad + \quad 3Ca(OH)_2 \qquad (1.1)$$

Tricalcium silicate water calcium silicate hydrate calcium hydroxide

Hydration of dicalcium silicate ($2CaO.SiO_2$):

$$2(2CaO.SiO_2) \quad + \quad 4H_2O \quad \rightarrow \quad 3CaO.2SiO_2.3H_2O \quad + \quad Ca(OH)_2 \qquad (1.2)$$

Dicalcium silicate Water Calcium silicate hydrate Calcium hydroxide

The hydration reactions result in calcium silicate hydrate and calcium hydroxide. Calcium silicate hydrate is adhesive and binds solid particles into a compact whole. It is the hydration product, largely, responsible for the strength gain of concrete (Soroka, I. 1979, **pp. 7, 31**).

The physical characteristic of cement particles which optimizes the rate, and amount of hydration, is fineness. Increasing fineness increases the surface area per unit mass of cement which means more contact surface area between water and cement particles is available. This increases the rate of hydration and consequently the rate of strength development. High degree of fineness also greatly reduces the diameters of the cement particles. The size of cement particles in ordinary Portland cement ranges from 0.005 mm to 0.055 mm (Soroka, I. 1979, **p. 37).** Hydration depends on availability of water. However, even when sufficient water is present, hydration stops after a time, although an appreciable amount of unhydrated cement is left in the cement grain core. This amount of unhydrated cement in the grain core may even exceed 50 % of the original weight of the unhydrous cement (Soroka, I. 1979, **p. 37).** The hydration process of a cement grain starts from the surface towards the inner core. This results in a building up of a layer of hydration products around it. In the presence of that layer, further hydration involves the diffusion of water through the layer. The rate of diffusion is partly controlled by the thickness of the layer; the greater the thickness, the slower the diffusion rate. When the thickness of the layer reaches 0.025 mm, the diffusion and the resulting hydration completely stop (Soroka, I. 1979, **p. 37).** Therefore, an unhydrated core is always present in a cement grain which has a diameter greater than 0.050 mm. For this reason, fineness of cement greatly influences the amount of hydration; the finer the particles, the greater the amount of hydration. In addition, other than for strength requirements, cement in concrete is used to provide workability on account of its high level of fineness (Schutz, R. J. 1980, **p. 9 – 24**).

Fineness is therefore an important property of cement. It may be expressed in terms of specific surface of cement particles (m^2 per unit mass) or the number of particles per unit mass. Fineness of ordinary Portland cement is of the order of 350 m^2/ kg (Blaine fineness) or 1.1 x 10^{12} particles / kg (Neville A.M. 2011, **pp.7, 71**). It is also usual to specify the fineness of cement particles in terms of sieve sizes. For ordinary Portland cement, 100 % of the particles should pass 0.150 mm aperture sieve and over 95 % of the particles should pass 0.075 mm aperture sieve (Kong, F.K., Evans, R.H. 1985, **p. 19**).

Despite the positive contribution of cement fineness to rate of strength development, amount of hydration, and workability of the concrete mixes, the fine state of division of cement particles gives them a tendency to flocculate in wet concrete. The flocculation entraps certain amount of water used in the mix and therefore all the water is not freely available to lubricate the aggregate particles. To disperse the flocculated cement particles, a plasticizing admixture (dispersant) is required. Plasticizers are substances that contain negatively charged molecules that get adsorbed onto the cement particles resulting in particle to particle repulsion. The overall results being deflocculated and dispersed cement particles. When this happens, the mixing water trapped within the flocs gets released and now becomes available for lubricating the aggregates leading to increased workability. The particle dispersion also eliminates the inter floc and inter particle friction to further increase workability. In the flocculated state, particle to particle contact faces are not readily available for hydration. Dispersion of the particles not only removes this limitation on hydration but also creates an even distribution of cement particles throughout the body of concrete by eliminating pockets of cement particle deficiency. This leads to higher concrete structural element strength. Plasticizers do not alter the hydration process or the nature of the hydration products (Shetty, M. S. 2009, **pp. 126 - 129**).

Sodium Lignin sulphonate, and calcium lignin sulphonate are examples of plasticizers in commercial use. They are basically water soluble plant extracts in which hemicellulose has been reduced to a low level. Plant biomass is made of cellulose (plant fibre), hemicelluloses (plant sugar), lignin, and extractives. In general, the first three compounds make up more than 90 %, by mass, of the plant material (Li_ Jingjing 2011, **p. 6**).The pine tree bark extract used in this study was expected to contain all the compounds except cellulose. No attempt was made to take the extract through the costly procedure of reducing the hemicellulose content. The purpose was to investigate whether or not, a local plant extract, obtained through a cost effective process can make a viable concrete plasticizing admixture. This was rigorously proved to be possible.

2. Literature review

2.1 Aggregates

Aggregates in concrete are used, primarily, for the purpose of providing bulk to the concrete. They provide about

75% of the body of the concrete (Shetty, M. S. 2009, **p. 66**). Coarse aggregate has maximum and minimum sizes of 40 mm and 5 mm, respectively, while those for fine aggregates are 5 mm and 0.075 mm, respectively (Neville, A.M. 2011, **p. 167**). In practice, the shapes of aggregates are described as rounded, irregular or angular (Kong, F.K., and Evans, R.H. 1985, **p. 23**). Naturally occurring aggregates are rounded or irregular and crushed ones are angular in shape. For the purposes of mix design, the only aspect of aggregates that is considered significant in affecting the workability of the fresh mix is whether the aggregate is crushed or uncrushed. Crushed aggregates result in lower workability (Building Research Establishment, Second edition, **p. 2**).

2.2 Acacia (Hashab: Arabic) tree extract

Use of plant extracts in concrete as admixtures has been studied by many scholars. Gum Arabic (Acacia tree extract) was used as an admixture in concrete, at the various dosage levels, expressed as a mass percentage of cement content. Both powder and liquid forms of the extract were studied. The research findings were that, for both physical forms of the extract, the significant dosage was 0.4 % of the cement content, a reduction in compressive strength, proportional to the dosage, occurred in both cases, and that Gum Arabic in liquid state, gave increased values of slump of fresh concrete mixes (Abdeljaleel, N. S. 2012**, pp. 57 - 65**).

2.3 Coconut plant extract

A study on extraction of sodium lignin sulphonate from coconut coir pith has been made. In the study, pith was extracted from coconut husks and then digested in an autoclave at 115 ^{o} C for 30 minutes and then the temperature was raised to 135 ^{o} C for 90 minutes. Before putting the material into the autoclave it was mixed with 2 % solution of sodium bisulphite. The coir pith material to solution mass ratio was 1:10, respectively. The pH of the mixture was maintained at an acidic range by use of dilute hydrochloric acid.

In this process, lignin in the pith reacts to form solid lignosulphonic acid which then dissolves in the black liquor. In solution, it combines with sodium to form soluble sodium lignin sulphonate. The sodium lignin sulphonate extract was concentrated under low pressure using a Rota Vap Aspirator Model A-35. A 325 ml sample of the concentrated black liquor yielded 6.0 g of sodium lignin sulphonate powder (1.85 %). Uses of sodium lignin sulphonate include making of concrete plasticizers (Sarma, U.S., and Rabindranath, A. D. 2003, **pp. 1 – 8**).

2.4 Indian Bamboo (*Bambusa Arundinacea*) extract

In a research involving this plant, the effectiveness of an extract from it as a concrete reinforcing steel corrosion inhibitor was studied. For the study, four concrete mixes A, B, C, and D were involved. A normal concrete mix design was carried out in accordance with Department of Environment method (U.K.). Materials output summary from that design is given in Table 2.1. The characteristic strength was 30 N/mm^2, ordinary Portland cement was used, and workability was of the range 30 mm to 60 mm slump. These materials quantities were used for all the mixes.

Table 2.1: Normal concrete mix design output summary

Cement content (kg/m^3)	Water content (kg/m^3)	Fine aggregates content (kg/m^3)	Coarse aggregates content (kg/m^3)
511	230	623	1016

(Abdulrahman, A. S. 2011, **p. 3)**

In concrete specimens from all mixes, reinforcing steel bars were embedded during casting. Mix A was used as a control. During mixing magnesium chloride ($MgCl_2$) was introduced into mixes B, C, and D to induce corrosion in the embedded steel. The mass of magnesium chloride added was 1.5 % of the mass of the cement content. Calcium nitrite $Ca(NO_2)_2$, a corrosion inhibitor in concrete, was added into mix C. The quantity of $Ca(NO_2)_2$ added was 2 % of the mass of cement content. Into mix D was added a plant extract from Indian Bamboo (*Bambusa Arundinacea*). The mass added was 2 % of the mass of cement content.

In the preparation of the plant extract, fresh leaves of Indian Bamboo were washed under running tap water, shade dried, and ground into powder. Each sample of powdered leaves, weighing 5 g, was soaked in 200 ml of 95 % ethanol solution for 14 days and thereafter filtered. In order to leave the sample free of ethanol, the filtrate was subjected to evaporation using a rotary evaporator. The concrete casting was in 100 mm cube moulds. Demoulding was after 24 hr and wet curing in sea water for 28 days at a temperature of 28^{o} C followed, for specimens from mixes B, C, and D. Control concrete (mix A) was cured in a potable water bath for 28 days. Corrosion of the embedded metal was measured after 180 days of exposure to wet and dry cycles. The measurements were done using electrochemical impedance spectroscopy and linear polarization resistance.

The conclusions were:

1.Comparison of corrosion observations from mixes A, B, C, and D confirmed that corrosion had been initiated in the metals embedded in mixes B, C, and D and that no corrosion had occurred in metal embedded in mix A.

2.Comparison of corrosion observations from mixes B, C, and D revealed that corrosion was inhibited in mixes C and D and that the plant extract (added to mix D) was a more effective inhibitor of metal corrosion than calcium nitrite incorporated in mix C (Abdulrahman A. S. 2011, **pp. 6484 – 6489**).

3. Methodology

3.1 Aggregates

Coarse aggregates used in this work were crushed and constituted from 10 mm and 20 mm single sizes in the mass ratio of 1:2, 10 mm: 20 mm single sizes, respectfully. The fine aggregates were uncrushed (river sand) and the percentage passing 0.600 mm sieve, determined by the sieve analysis test, was 55 %. Water absorption for aggregates was determined in accordance with BS882. For fine aggregates, moisture contained in saturated and surface dry (SSD) aggregates was found to be 0.92 %: typical values are in the range 0.20 % to 3.15 %. Same test on coarse aggregates, gave 3.82 % for absorption while typical values are in the range 0.80 % to 4.53 % (Neville, A.M. 2011, **p. 131**).

3.2 Pine bark extract preparation

Fresh pine bark was cut into pieces of convenient size. The material was then put into a digester into which tap water was added. The mass ratio of bark: water was 1: 2 respectively. The temperature of the digester was then raised from the ambient level to 121^{o} C in 15 minutes. At this temperature the pressure attained in the digester was 0.1 N/mm^2. These conditions of heat and pressure were maintained for one hour after which period the water had turned opaque brown (black liquor). The separation of black liquor and the bark was effected by filtration through a muslin cloth bag.

3.3 Design of normal concrete mix (control)

Design for characteristic strength class 20 concrete, was in accordance with the current Building Research Establishment (BRE), method. This procedure uses the equation:

$$f_m = f_c + M \qquad (3.1)$$

Where f_m = the target mean strength, f_c = the specified characteristic strength, and M = the margin = ks: where k = a value appropriate to the percentage defectives adopted, derived from the mathematics of normal distribution. The value of k increases as the proportion of defectives is decreased. The k value for 5 % defectives is 1.64. This is the value specified by BS 8110 and was adopted for this research work (BS 8110: Part1: **clause 2.4.2.1**). s = standard deviation. The numerical value of s was taken as 8 (BRE, figure 3, **p. 12**).

The maximum cement content was restricted to 550 kg/m^3 (Kong, F.K., Evans, R.H. 1985, **p. 39**). For durability, moderate exposure conditions were assumed, resulting in a minimum cement content of 290 kg/m^3 (Murdock, L.J., and Brook, K. M. 1979, **p. 121**). The water/cement ratio was unrestricted. The slump value range for the mix was taken as 30 mm to 60 mm for design. During casting, the slump was further restricted to 45 $\pm$ 5 mm. Cement contents for three trial mixes were 315 kg/m^3, 365 kg/m^3, and 400 kg/m^3 (BRE, **p. 17**). A cement content of 400 kg/m^3 proved satisfactory. Summary of material quantities is presented in Table 3.1.

Table 3.1: Mix design summary

Quantities	Cement (kg)	Water (kg or litres)	Fine aggregates (kg)	Coarse aggregates (kg)		
				10mm	20 mm	40mm
Per m^3	400.0000	242.1886	668.8466	400.8093	801.1555	-

3.4 The test concrete mixes

Each of the test mixes had the same material quantities as the control mix except for water which was partially substituted with pine bark extract. Dosages of the black liquor used, expressed as a mass percentage of the cement content were 10 %, 20 %, 30 %, 40 %, and 47.5 %. In every case, the mixing water was reduced by an amount equal to the mass of the dosage. Compressive strength tests were carried out at the ages of 3, 7, 28, and 61 days. As for the control concrete, the workability of the concrete was held constant at 45 $\pm$ 5 mm slump. For the test mix with plant extract at a dosage of 40 %, further tests of flexural, and cylinders splitting tensile strength were done. To assess the effect of the extract on the workability, mixes were made with the water/cement (w/c) ratio held constant at 0.6055. The pine bark extract dosage and the mixing water were varied as in the cases for testing for compressive strength. Slump tests were used to assess the workability of the concrete and values reported to the nearest 5 mm.

3.5 Concrete casting

Before adding cement and plant extract into the mix, the weighed aggregates were mixed with part of the mixing water and 5 minutes allowed to elapse before proceeding with the mixing operation. This fraction of the mixing water was at least the amount required to bring the aggregates to SSD condition. This procedure had the effect of minimizing the amount of plant extract getting into the pores of the aggregates and thus optimized its effects on strength and workability.

Casting was done in100 mm cube moulds. Cylinder moulds' measurements were 100 mm diameter x 200 mm height while the beam sizes were 100 mm x 100 mm x 500 mm long. Since the maximum coarse aggregate particle size was 20 mm these sizes met the requirements of BS1881: Part 3. Demoulding of the samples was done 24 hours after casting followed by water bath curing till testing age.

4. Experiments, results, and discussions

4.1 Determination of water absorption of coarse aggregates

The samples were washed clean and soaked in water for 24 hours. The aggregates were separated from the water by decanting. Surface moisture on the particles was wiped out using an absorbent dry cloth and then transferred onto a second absorbent dry cloth. The particles were then allowed time for the moisture to dry out away from sun's radiation. The particles changed colour when they attained saturated surface dry condition. In this state the mass A (g) was taken. The aggregates were then transferred into a graduated plastic cylinder and water added to a particular volume such that the aggregate particles were fully submerged. The mass of the aggregates, flask and water B (g) was taken. By decanting, the aggregates were separated from water. The measuring cylinder was then filled with water only, to the same volume and the mass C (g) taken. Wet aggregates were then dried in an oven at 110 ^{o}C for 24 hours and then mass of oven dried aggregates D (g) taken. The results are given in Table 4.1.

Table 4.1: Determination of water absorption of coarse aggregates

	Sample mass (g) (10 mm – 5 mm)			Sample mass (g) (20 mm – 10 mm)		
	I	II	III	I	II	III
Mass of sample in SSD condition - A	1037.0	1042.0	1015.0	1050.0	1015.5	1042.0
Mass of aggregates + water + pycnometer - B	1606.0	1610.0	1591.0	1830.0	1812.0	1825.0
Mass of water + pycnometer – C	982.0	982.0	982.0	1199.0	1199.0	1199.0
Mass of oven dried sample – D	978.5	1002.5	1000.5	1015.0	977.5	1000.0
Water absorption (%) = (A – D)100/(D)	5.98	3.94	1.45	3.45	3.89	4.20
Mean absorption (each size) = (I +II + III)/(3)	3.79			3.85		
Mean absorption for both sizes (%)	3.82					

4.2 Determination of water absorption of fine aggregates **(**5 mm – 0.075 mm)

Samples of river sand, sieved through 5.0 mm sieve and retained on 0.075 mm sieve weighing about 500 g were washed clean over a 0.075 mm sieve and then soaked in water for 24 hrs. The wet sand was separated from water by decanting over 0.075 mm sieve. The wet sand was placed on a non absorbent surface and the surface moisture allowed to dry out, avoiding direct sun's radiation. When the saturated surface dry (SSD) state was reached, the sand changed colour. The sand was then filled into an inverted flow test cone in three layers. Each layer was compacted by giving it 25 blows using a standard tamping rod . The rod was raised 25 mm above the surface of the sand and then allowed a free fall onto the sand, to constitute a single blow. After compaction of the third layer, the top of the truncated cone was leveled off and the sand around the base removed. The cone was then lifted, vertically, off the sand. Shearing of the sand cone indicated that the sand was at the SSD state. None shearing indicated that the sand was still wet and needed more time to dry out. Collapse of the cone indicated dry sand and the test would be repeated. Moisture content of sand from the sheared cone was taken as the water absorption of the fine aggregates. The results are given in Table 4.2.

Table 4.2: Determination of absorption of fine aggregates

	Sample mass (g)		
	I	II	III
Mass of sand sample at SSD state - A	526.0	556.0	514.0
Mass of dry sample - D	520.0	552.0	509.0
Water absorption (%) = (A – D)100/(D)	1.154	0.63	0.98
Mean percentage water absorption = (I +II + III)/3	0.92		

4.3 Determination of the grading of the fine aggregates

For the purposes of normal concrete mix design, in accordance with British Research Establishment (BRE) method, fine aggregates are graded on the basis of the percentage of the mass passing the 0.600 mm sieve. The sieve analysis test was conducted on three representative samples of sand, each weighing about 500 g. Results are given in Table 4.3: mean percentage passing the 0.600 mm sieve expressed to the nearest 5 %.

Table 4.3: Results of sieve analysis test

	Sample mass (g)		
	I	II	III
Mass retained on the 0. 600mm sieve but passing 5.0 mm sieve (A)	221.0	251.5	243.0
Mass passing the 0.600 mm sieve (B)	298.0	304.0	269.0
Total mass of the sample (C) = (A+B)	519.0	555.5	512.0
Percentage mass passing the 0.600 mm sieve = (B)(100)/(C)	57.42	54.73	52.54
Average percentage mass passing the 0. 600 mm sieve = (I + II + III)/3	55		

4.4 Results of experiments on fresh and hardened concrete

4.4.1 Compressive strength tests

Typical results are presented in Tables 4.4 to 4.7 and summarized in Table 4.8. Graphical presentation of these results is given in Figures 4.1, and 4.2.

Table 4.4: Compressive stress and slump values of control concrete (0.0 % pine bark extract admixture)

Age (days)	Area (mm^2)	Load (kN)	Stress (N/mm^2)		w/c ratio	Slump (mm)	
			Sample stress	Mean stress		Actual	Target
3	1000	173.646	17.5	18.0	0.6055	45	45 ± 5
		174.346	17.5				
		186.665	18.5				
7	1000	313.718	31.5	31.0			
		317.091	31.5				
		292.800	29.5				
28	1000	410.912	41.0	41.0			
		395.788	39.5				
		432.312	43.0				
61	1000	507.689	51.0	48.0			
		444.933	44.5				
		485.407	48.5				

Table 4.5: Compressive stress and slump values of concrete containing admixture: pine bark extract (dosage 10 %)

Age (days)	Area (mm^2)	Load (kN)	Stress σ (N/mm^2)		w/c ratio	Slump (mm)	
			Sample stress	Mean stress		Actual	target
3	1000	251.391	25.0	25.5	0.5937	40	45 ± 5
		252.736	25.5				
		259.796	26.0				
7	1000	361.203	36.0	32.0			
		311.280	31.0				
		296.553	29.5				
28	1000	465.054	46.5	47.5			
		507.796	51.0				
		445.352	44.5				
61	1000	509.257	51.0	48.5			
		464.755	46.5				
		478.072	48.0				

Table 4.6: Compressive stress and slump values of concrete containing admixture: pine bark extract (dosage 20 %)

Age (days)	Area (mm^2)	Load (kN)	Stress σ (N/mm^2)		w/c ratio	Slump (mm)	
			Sample stress	Mean stress		Actual	Target
3	1000	254.137	25.5	24.0	0.6055	50	45 ± 5
		210.795	21.0				
		250.495	25.0				
7	1000	334.021	33.5	34.5			
		357,585	36.0				
		337.038	33.5				
28	1000	479.725	48.0	47.5			
		485.118	48.5				
		463.368	46.5				
61	1000	547.316	54.5	54.5			
		537.379	53.5				
		554.662	55.5				

Table 4.7: Compressive stress and slump values of concrete containing admixture: pine bark extract (dosage 40 %)

Age (days)	Area (mm^2)	Load (kN)	Stress (N/mm^2)		w/c ratio	Slump (mm)	
			Sample stress	Mean stress		Actual	Target
3	1000	251.055	25.0	25.5	0.5905	45	45 ± 5
		266.408	26.5				
		252.624	25.5				
7	1000	255.678	25.5	27.5			
		264.587	26.5				
		305.140	30.5				
28	1000	514.415	51.5	48.0			
		399.690	40.0				
		521.766	52.0				
61	1000	515.386	51.5	52.0			
		513.993	51.5				
		532.074	53.0				

Table 4.8: Average stress values at different ages and the relevant slump values

Pine bark extract as a mass percentage of the cement content	Mean compressive stress (N/mm^2)				w/c ratio	Water reduction (%)	Slump (mm)	
	3 days	7 days	28 days	61 days			Actual	Target
0.0	18.0	31.0	41.0	48.0	0.6055	0.0	45	45 ± 5
10.0	25.5	32.0	47.5	48.5	0.5937	2.0	40	
20.0	24.0	34.5	44.5	54.5	0.6055	0.0	50	
30.0	24.5	30.5	46.0	48.5	0.6055	0.0	40	
40.0	25.5	27.5	48.0	52.0	0.5905	2.5	45	
47.5	23.0	33.5	43.5	48.0	0.5849	3.4	50	

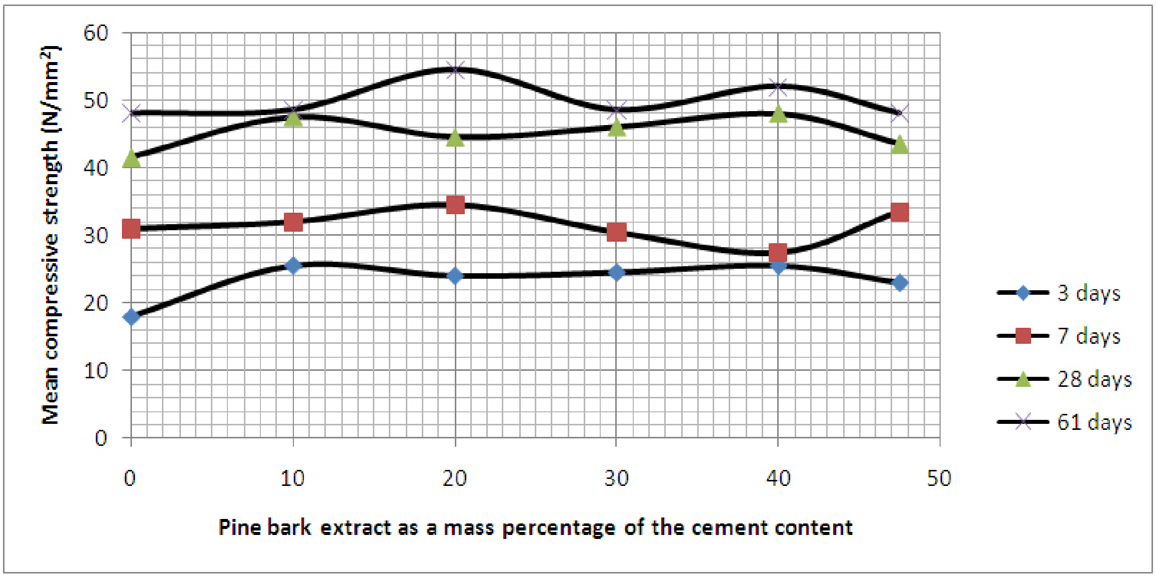

Figure 4.1: Relation between pine bark extract dosages and the mean compressive stress at ages 3, 7, 28, and 61 days.

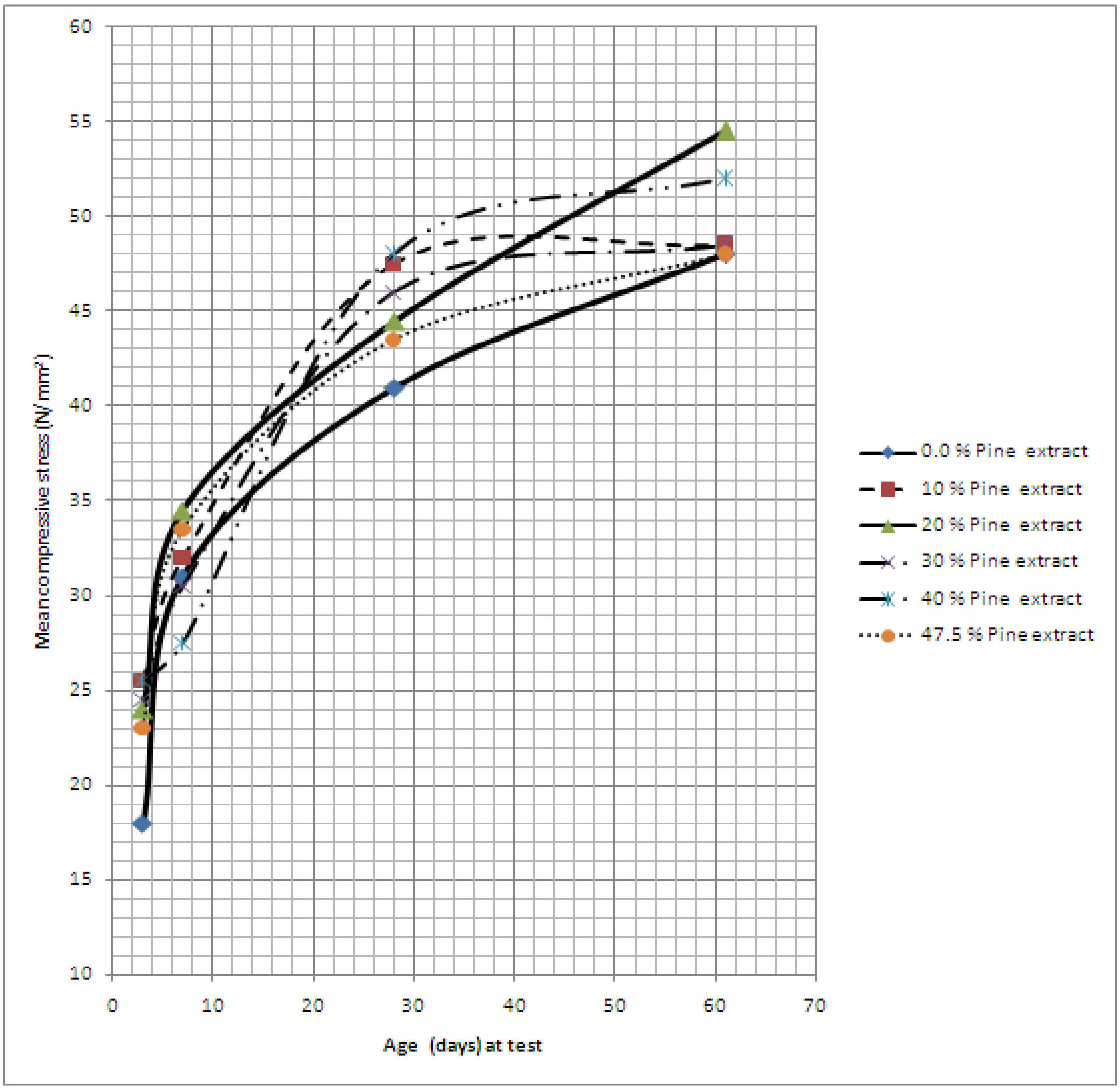

Figure 4.2: Variation of strength with age for concrete containing pine bark extract admixture at varying dosages.

As illustrated in Figure 4.2, at the plant extract dosage of 20 %, the stress values were higher than those of control concrete at all the ages of testing. The stress increases were 33.5 %, 11.5 %, 8.5 %, and 13.5 % at 3, 7, 28, and 61 days, respectively.

4.4.2 Effect of the pine bark extract admixture on the mixing water content

The observed concrete mixing water reduction values, due to the use of the pine bark extract admixture, and the corresponding dosages of the admixture are presented in Table 4.8 and plotted in the scatter diagram Figure 3. Visual inspection of the figure suggests a positive linear relationship between the two sets of data. The strength of that relationship is measured by the linear correlation coefficient r. When r = +1, all the data points perfectly fall on a straight line with a positive gradient to the right, and r = 0 when there is no linear relationship between the two sets of data (Walpole, R.E. 1986, **pp. 333 – 345)**.

The number of different dosages used in the experiment n = 6. If pine bark extract dosage is x, and the corresponding mixing water percentage reduction is y:

$$Linear\ correlation\ coefficient\ r\ = \frac{n\sum xy - (\sum x)(\sum y)}{\sqrt{[n\sum x^2 - (\sum x)^2][n\sum y^2 - (\sum y)^2]}} \tag{4.1}$$

Table 4.9: Computations for the linear correlation r and linear regression

							$\sum_s$
Pine bark extract (%) dosage (x)	0	10	20	30	40	47.5	147.5
Mixing water (%) reduction (y)	0	2.0	0.0	0.0	2.5	3.4	7.9
xy	0.0	20.0	0.0	0.0	100.0	161.5	281.5
x^2	0.0	100.0	400.0	900.0	1600.0	2256.25	5256.5
y^2	0.0	4.0	0.0	0.0	6.25	11.56	21.81

Substituting numerical values, from Table 4.9, in equation (4.1):

$$Linear\ correlation\ coefficient\ r\ = \frac{(6)(281.5) - (147.5)(7.9)}{\sqrt{[(6)(5256.5) - (147.5)^2][(6)(21.81) - (7.9)^2]}}$$

$$= \frac{1689\ - 1165.25}{\sqrt{[31539\ - 21756.25][130.86\ - 62.41]}} = \frac{523.75}{\sqrt{[9782.75][68.45]}} = \frac{523.75}{818.3087666}$$

$$= 0.64004\ = 0.640$$

When tested at 5 % level of significance, the value for r should at least be equal to 0.729. Therefore, the linear relationship between the two sets of data is not strong. The shape of the aggregates could also have influenced the relationship but is not accounted for in the experimental data analyzed.

Determination r^2 = 0.41. Hence, 41 % of the results is what can be accounted for by linear relationship. (Walpole, R.E. 1986, **pp. 339– 341, 408)**.

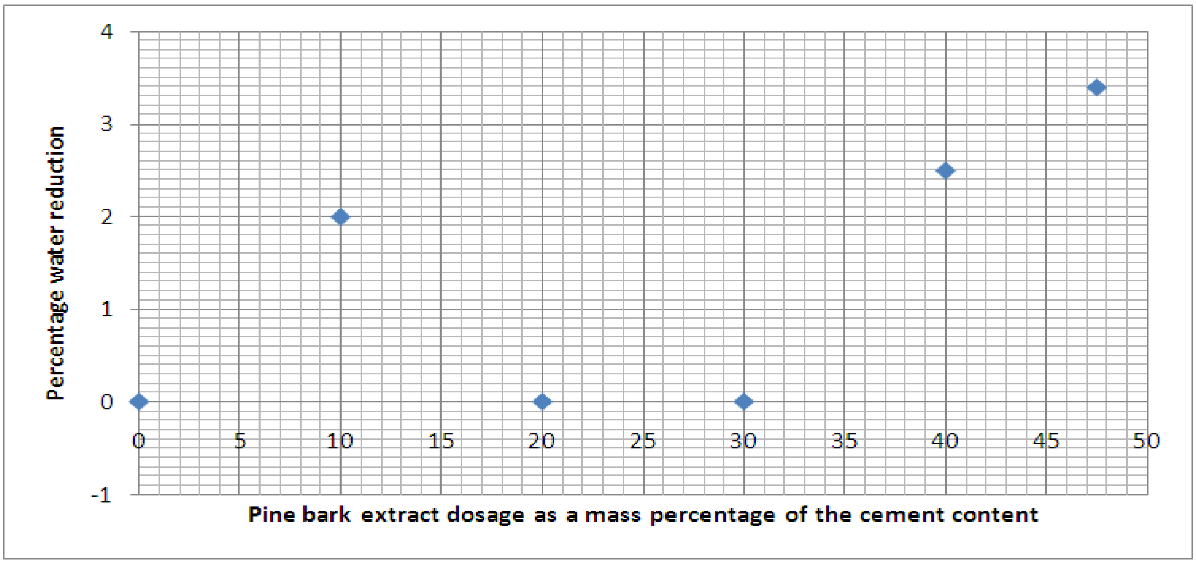

Figure 4.3: Scatter diagram

Linear regression
Linear relationship between the mixing water percentage reduction and the dosage of the pine extract admixture may be defined by the equation:

$$Percentage\ water\ reduction\ y = a + bx \quad (4.2)$$

$$where\ b = \frac{n\sum xy - (\sum x)(\sum y)}{n\sum x^2 - (\sum x)^2} \quad (4.2.1)$$

$$and\ a = \frac{\sum y - b\sum x}{n} \quad (4.2.2)$$

Substituting numerical values, from Table 4.9, in equations (4.2.1), and (4.2.2):

$$b = \frac{(6)(281.5) - (147.5)(7.9)}{(6)(5256.5) - (147.5)^2} = \frac{1689 - 1165.25}{31539 - 21756.25} = \frac{523.75}{9782.75} = 0.0535$$

$$a = \frac{7.9 - (0.0535)(147.5)}{6} = 0.0015$$

Hence, substituting the numerical values of the constants a and b in equation (4.2):

$$Percentage\ water\ reduction\ y = 0.0015 + 0.0535x \quad (4.3)$$

Where x is the pine tree bark extract, expressed as a mass percentage of the cement content (Walpole, R.E. 1986, **pp. 333 – 345**).The plot is given in Figure 4.4.

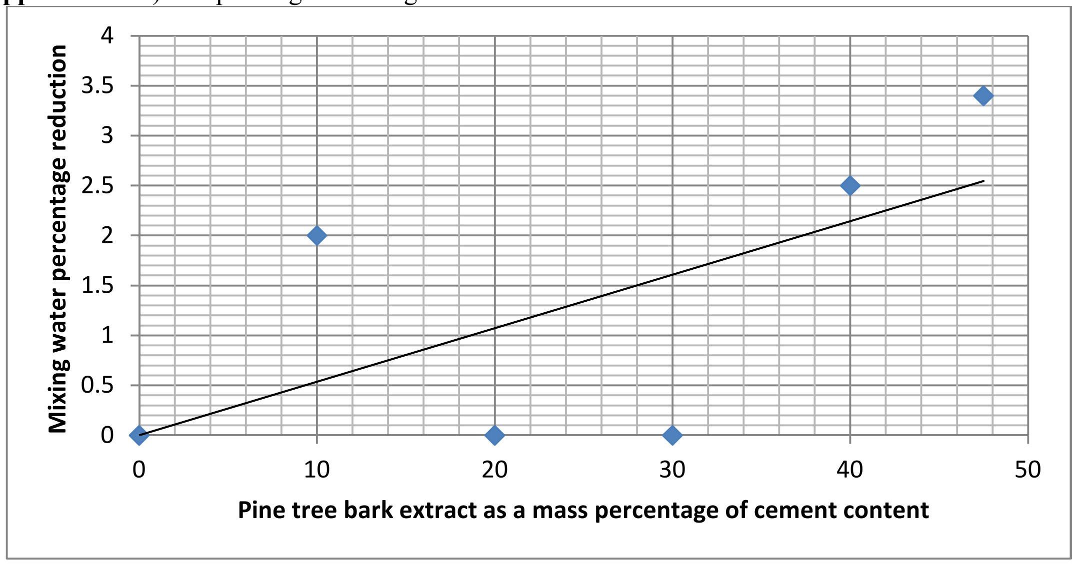

Figure 4.4: Variation of concrete mixing water reduction percentage with the dosage of pine bark extract admixture

4.4.3 Workability tests.
Table 4.9: The slump test values, obtained with different dosages of the pine bark extract.

Pine bark extract as a mass percentage of the cement content	w/c ratio	Slump (mm)
0.0	0.6055	45
10.0	0.6055	155
20.0	0.6055	160
30.0	0.6055	165
40.0	0.6055	140
47.5	0.6055	150

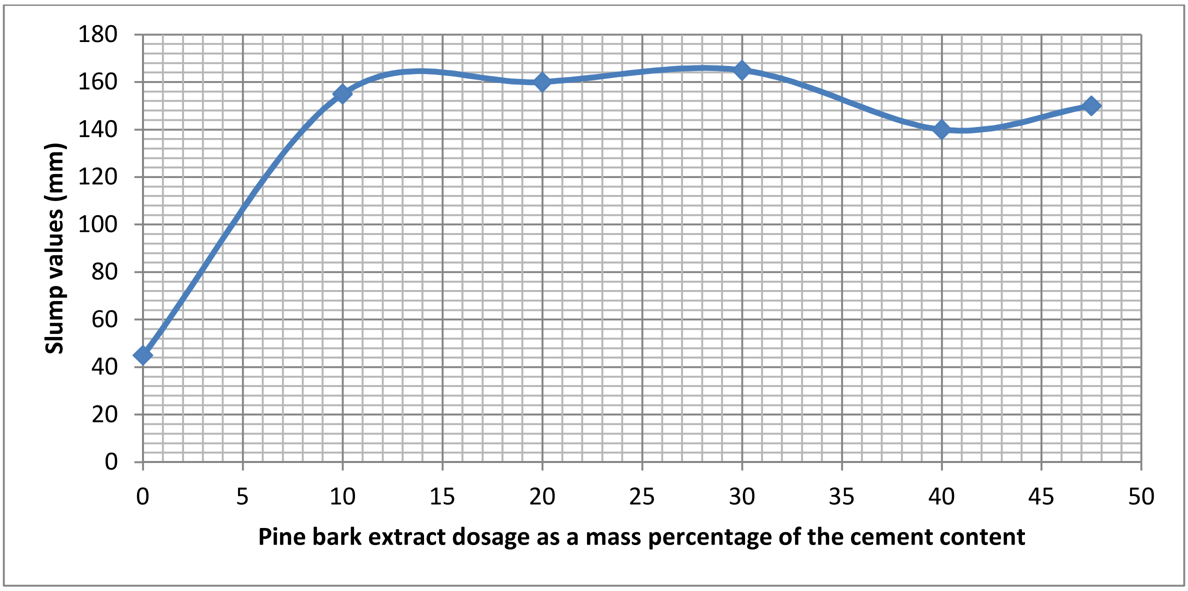

Figure 4.5: Variation of slump tests values with the pine bark extract dosages

4.4.3 Indirect tensile strength (cylinder splitting tensile strength) tests

Table 4.10: The 28 days cylinder splitting strength values (40 % dosage of the pine bark extract)

Pine extract dosage	Cylinder sample mass (g)	Load (kN)	The 28 days cylinder splitting strength (N/mm^2)	
			Sample stress	Mean stress
40 %	3713.5	90.822	2.891	3.0
	3746.5	88.631	2.821	
	3730.5	88.297	2.811	

The cylinder splitting strength of concrete is 8.3 % to 12.5 % of its compressive strength (Kong, F.K., and Evans, R.H. 1985, **p.26**). From Table 4.8, the 28 days compressive strength of this concrete is 48.0 N/mm^2 whose 8.3 % is 4.0 N/ mm^2.

4.4.4 Flexural strength tests (Two points loading).

The flexural strength of concrete is about 150 % of its cylinder splitting strength (Kong, F.K., and Evans, R.H. 1985, **p.27**). From Tables 4.10 and 4.11, this relationship between the two types of stress is unaffected.

Table 4.11: The 28 days flexural stress values (40 % dosage of the pine bark extract)

Pine extract dosage	Sample mass (g)	Crack eccentricity (mm)	Load (kN)	Flexural strength (N/mm^2)	
				Sample stress	Mean stress
40 %	12191.5	75	9.761	4.393	4.5
	12471.0	35	9.095	4.093	
	11981.5	40	9.944	4.475	

5. Conclusions and recommendations

Pine tree bark was harvested from a living tree and using water of potable quality, heat and pressure, an extract was made. Without further processing, the extract was used as an admixture in concrete at dosage levels of 10 %, 20 %, 30 %, 40 %, and 47.5 % of the cement content. The compressive strength of concrete was tested at ages 3, 7, 28, and 61 days. For all the mixes, the casting of the concrete was done to achieve the same level of workability of 45 mm slump; with a tolerance of 5 mm. The stress values so obtained are presented in Table 4.8, and graphically in Figures 4.1, 4.2, and 4.2.1. Compressive strength of concrete was found to consistently increase, at all the ages tested when the admixture dosage was 20 %.

Mixes to assess the effect of the extract on the workability of concrete were also cast but with the w/c ratio fixed at 0.6055. Values of the slump tests results are presented in Table 4.9 and graphically in Figure 4.5. From these results the extract admixture increased the workability of the fresh mixes. The increase was directly proportional to quantity of extract used until a dosage of about 15 % was reached at which point a maximum increase was attained. At this peak value, workability of the concrete containing the extract admixture was about

3.5 times the workability of the control concrete. This peak value remained constant even after increasing the dosage.

Effects of the admixture on the cylinder splitting (indirect tension), and flexural strengths were also investigated at the dosage of 40 %. The tests were carried out at the age of 28 days. From Table 4.8, the compressive strength attained at that age was 48.0 N/mm^2. From Table 4.10, the cylinder splitting strength obtained was 3.0 N/mm^2 which is 6.3 % of the compressive strength. It was expected to fall within the rage 8.3 % to 12.5 %. From Table 4.11, the flexural strength of this concrete at 28 days was 4.5 N/mm^2. Flexural strength is supposed to be about 150 % of the cylinder splitting strength (Kong, F.K., and Evans, R.H. 1985, **pp. 26 - 27**).

It can be concluded that pine tree bark extract concrete admixture, prepared as hitherto explained will:

1) At a dosage of 20 %, by mass, of the cement content, increase the strength of concrete at all ages covered in this study. These increases will be of the order of 33.5% at 3 days, 11.5 % at 7 days, 8.5 % at 28 days, and 13.5 % at 61 days.
2) Increase workability of the fresh concrete, at all the dosages covered in this study and at a dosage of 20 %, by mass, of the cement content, the slump value will increase by a factor of about 3.5.

From the findings of this research, it can be recommended that pine tree bark extract, specifications as described above, at a dosage of 20 %, by mass, of the cement content can be used as an alternative to the concrete plasticizing admixtures.

6. Acknowledgements

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