Curing and Hydration in Cement - Aluminum Dross Concrete Mortar

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Abstract

Cementitious-paste matrix-bond, curing method, and curing period are important factors affecting concrete compressive strength, hence this study focused on the evaluation of the concrete phase transition from the fresh to hardened stage and the characteristics of bond strength of solid objects when aluminum dross (AlDr) is used as partial replacement in cement concrete. Setting times period of AlDr-Cement were examined, and the hydration process commences with adding water to cement to form cement paste until the paste is set and plastic or viscoelastic, in preparation for the curing and hardening process. Similarly, AlDr-Cement mortar (75x75x75mm) cubes were produced and exposed to air, laboratory water, and lagoon water curing for 28 days. Compressive strength tests at 7 days, 14 days, 21 days, and 28 days were performed. Observations indicated that setting time at 10 wt.% gave a better result than the rest with good adhesive binding properties and a setting period of 100 mins between the initial and final setting time of 180 mins**.** This defines the period for curing and hardening cementitious paste (or solidifying)**.** The paste became set through a chemical reaction of cement with moisture known as the hydration process. The mechanism entails the formation of Calcium Silicates hydrates and Tricalcium Aluminate hydrate as product and with time produces a solid and hard mass that binds aggregates in concrete. Compressive strengths were 8N/mm^2 max (0 wt.% AlDr) at 28 days air curing, 9.78N/mm^2 max. (10 wt.% AlDr) at 28 days of water curing and 6.67N/mm^2 max. (15 wt.% AlDr) at 21 days lagoon water cured. It was also identified that partial addition of AlDr replacement of cement is pozzolanic, with the conclusion that 10 wt.% AlDr-Cement is adequate for concrete mortar applications.

Keywords: Cement paste, Aluminum-dross, setting times, compressive-strength, pozzolanic

Introduction:

Concrete is a composite material of fine and coarse aggregates bonded with cement paste to form a homogeneous solid object. The cement paste is formed through a chemical reaction between cement and water (Moses and Perumal, 2016), and sometimes with other ingredients to form a cement matrix that binds aggregates firmly in concrete. Additives, such as pozzolan or super-plasticizers (Neville and Brooks, 2010), may be included in the mixture to improve the characteristic properties of fresh and hardened concrete. Concrete Mix design is an engineering process used to determine the proportioning of concrete constituents for specific concrete characteristic performance (Kumhar and Murnal, 2012), such as workability (i.e., consistency and stability), compressive strength, stress/strain performance, and durability. The constituent proportioning according to a specific mix design is known as the concrete mix ratio used in construction during the batching process for concrete production and in quality control. Pozzolans are siliceous or aluminous materials with little or no cementitious value, but in finely ground form in the presence of moisture react chemically with calcium hydroxide ($Ca(OH)_2$) to form compounds possessing hydraulic cementitious properties. According to Al-Chaar *et al.* (2013), pozzolans are materials that, when used in conjunction with Portland cement, will contribute to the properties of hardened concrete through hydraulic or pozzolanic activity. Concrete displays two distinct phases with different characteristics from the production to the hardened stage. Fresh concrete is plastic and fluid at manufacture, with no characteristic strength for engineering purposes and load bearing (Afolabi *et al.,*2021), but becomes hardened overtime during the curing and hydration process, which requires about 28 days for normal concrete to develop acceptable characteristic strength. Concrete becomes valuable and significant for load bearing at the hardened stage. The cement paste/binder is formed through chemical reactions (i.e., hydration) of Portland cement and water, which continues over time until the attainment of stable chemical equilibrium that binds the aggregates to produce hardened concrete occurs (Moses *et al.,*2016; Neville and Brooks, 2010). The reaction, although initially fast (i.e., setting and hardening of cementitious paste) continues for a longer period, to allow the formation of adequate intermolecular bonds between the cement paste and aggregates. The chemical bond is the attraction between atoms, ions, or molecules that enables the formation of chemical compounds, and bond development in concrete follows the transformation sequence from plastic bonds in fresh concrete, which later metamorphous to solid bonds of hardened concrete (Afolabi *et al.,*2021). The phase change from fresh (i.e., plastic phase) to hardened concrete is initiated by the hydration process of cement paste, through the curing process. Hydration of cement is the reaction, or series of chemical reactions, of cement with water to form the binding material of aggregates (Bullard *et al.,*2011). Thermodynamic equilibrium is an internal state of a single thermodynamic system or a relation between several thermodynamic systems connected by more or less permeable or impermeable walls. In thermodynamics equilibrium, there are no net microscopic flows of matter or energy within a system or between systems (Freidt, 2012). Chemical stability is the thermodynamic stability of a chemical system, which occurs when a system is in its lowest energy state or chemical equilibrium with its environment, and the

equilibrium will persist indefinitely unless the system is changed, e.g., change in the phase of matter or a set of chemical reactions. At chemical equilibrium or in phase equilibrium, the total sum of the product of chemical potentials and stoichiometric coefficients is zero, as the free energy is at aluminium (Schroeder, 2021). Chemical equilibrium is the state in which both reactants and products are present in concentrations that have no further tendency to change with time and occur when the forward reaction proceeds at the same rate as the reverse reaction and there are no net changes in concentration of the reactants and products, a condition also known as dynamic equilibrium. Transformation of freshly prepared mortar involves the change of state from plastic to hardened with corresponding changes in internal energy ΔU (difference of energy between initial and final state). Therefore, $\Delta U = \sum E$, and E = various energies transferred to the system from the reference state to the given state

1.1: Panditharadya*etal.* (2021), studied the effect of Aluminium Dross on workability and Setting Time of Cement and Concrete.Aluminum dross is rich in aluminium oxide and has traces of a few heavy metals, indicating pozzolanic characteristic behaviour. They investigated the behavior of aluminium dross as a partial replacement for Ordinary Portland Cement with replacement at 5, 10, 15, and 20% of Ordinary Portland Cement. The consistency and setting time of cement paste samples were determined, which shows a lag in the setting time of the paste. The effect of external curing methods on the development of mechanical and durability-related properties of normal-strength concrete was investigated by Atsbha and Zhutovsky (2022). The study expressed curing concrete as a critical step and must be applied appropriately for any concrete structure to develop the desired mechanical and durability-related properties. External curing methods provide extra water (water curing methods) or limit the loss of mixing water (sealed methods) from the mix, aiding the concrete to achieve the required properties. The experimental results indicated that 28 days of wet curing is the optimum approach, and to be considered acceptable, any curing method must produce concrete properties equivalent to 7-day wet curing. Akinwunmi and Gbadamosi (2014), evaluated and presented the results of an experimental study on the effects of curing methods and curing ages on compressive strength development of ordinary Portland cement concrete in a tropical environment. Fifteen (15) concrete cubes were cured by immersion respectively in potable water, immersion in lime water, covering with awet rug, covering with plastic sheets, and air-drying. For each of these curing methods, the average compressive strength of concrete cubes was determined after 3, 7-, 14-, 28- and 90-day curing periods. The result obtained does not encourage the use of curing by air-drying method and also suggested limiting the use of other curing methods to 28-days period. Generally, the highest compressive strength was obtained for concrete cured by immersion in lime water. Nahata *et al*. (2013), investigated the effect of curing on the efficiency of curing cement mortar and also evaluated the compressive strength of mortar cubes at 28 days, and the effect on strength with the application of different compounds. The study adopted the methods of structural grade mix with cement: sand ratio 1:2.75 and varying water/cement binder ratio between 0.45 to 0.60 using field sand, ASTM-graded sand and OPC. Comparing the compressive

strength results of different curing mechanisms indicated that using membrane curing compounds, an efficiency of 80-90% can be achieved as compared to conventional water curing.

Research Methodology and Materials

2.1. Concrete exhibits a phase transition from plastic to hardened concrete over a period, till when it must have developed adequate strength, which involves a change from loose bond plastic to solid bond in hardened concrete possessing adequate energy and bond strength, The strength of chemical bond varies considerably and depends on potential force of attraction (Frenking and Shaik, 2014) between interacting substances. The study focused on the evaluation of available bond strength in the cementitious paste matrix of concrete, by considering the requirement of chemical bond formation, phase transition from fresh to hardened concrete, setting period, and concrete curing (Lyman and Yuanbo, 2015). The setting is the period of transformation from fresh concrete to semi-solid that enables the commencement of curing and concrete hardening to a solid object possessing sufficient strength and properties for load application and engineering purposes.

The strengthening process of cement paste is as follows,

Cement paste → hydration → setting period → curing and hardening → load application

2.2: Experimental Materials: The concrete constituents' materials are, cement, fine aggregates (sand), and coarse aggregates (granite); aluminum dross (AlDr) sourced from a secondary smelting process and used as a partial replacement of cement (Afolabi, 2023). The powdered AlDr obtained from Aluminum Rolling Mills (Ota, Ogun State Nigeria) is a byproduct of the secondary smelting process with the chemical composition shown in Table 1. Preliminary and quality control tests were carried out on constituent materials, and the following tests were conducted at the University of Lagos Concrete Laboratory, Setting time tests on cement paste, casting of concrete mortar (75x75x75mm) cubes, and compressive tests on mortar cubes.

Table 1: Aluminum dross chemical composition

Constituent	SiO_2	CaO	Na_2O	Al_2O_3	Fe_2O_3	MgO	SO_3	K_2O	Al
Percent composition	7.15	0.07	0.06	63.84	0.03	0.04	0.03	0.01	28.77

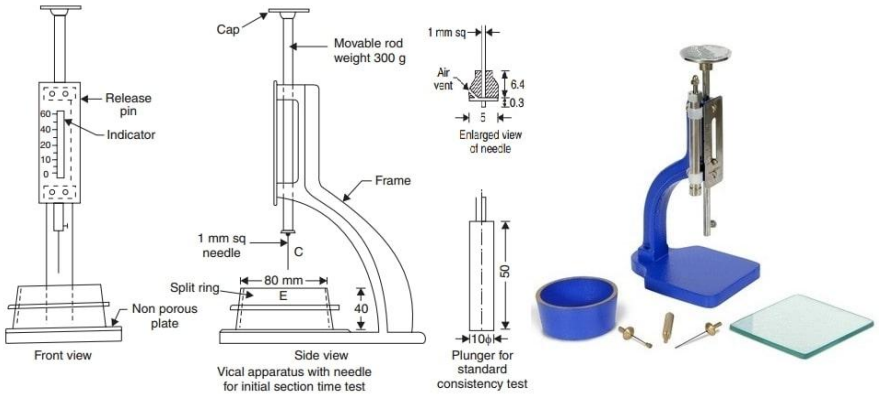


Figure 1: Vicat Apparatus

Setting Time Test: The time is implemented using ASTM C191-21; Standards Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle. The equipment includes vicat apparatus, weighing machine, stopwatch, gauging trowel, measuring cylinder and tray, etc. Initial setting time is the time required by the cement paste to acquire plasticity and commence transformation from the fresh stage, while final setting time is the time required to become fully plastic or visco-elastic material for the hardening process to commence, hence this transformation process can be supported by curing to enable the adequate formation of solid bonds.

Concrete Compressive Strength Test: Implemented according to ASTM C109; Standard Test Method for Compressive Strength of Hydraulic Mortars. The specimen cubes (75x75x75mm) were subjected to compressive loading and observed until collapse, the ultimate load at fracture was recorded. Compressive strength is defined as the stress (or, load point), at which the material fractures at the ultimate load, and characterized directly with material bond energy (or strength) and defined as the bond dissociation energy. Bond energy (E) is the measure of bond strength in a chemical bond (Deiaf, 2016) and is defined as an average of all bond-dissociation energies in a molecule since the energy required to break a “single bond” in a molecule differs for each bond in the molecule. Concrete bond is neither covalent nor ionic because the aggregates possess little or no electrochemical charge and affinity for chemical attraction required to form stable (Frenking and Shaik, 2014), but rather exhibit a partial covalent mechanism of adherence of the cement paste that bound the aggregates particles in concrete, which becomes effective and stronger during the curing process. The constituents (i.e., aggregates) are nonbonding because they are stable molecules but the chemical reaction of cement and water produces a compound (cement paste) with adhesive properties and characteristics that bind the aggregates in concrete formation.

Results and Discussion

3.1: Mortar Initial and Final Setting Times Characteristics

The initial setting time is the commencement time of the transformation of fresh cementitious paste to plastic. In contrast, the final setting is the time that elapses when the cement paste has completely transformed to plastic material. At this stage, the Vicat needle cannot penetrate the material due to the hardness property developed. In Table 2 the test results indicated that 10wt. % of AlDr had an early setting time and the least final setting time compared with the control (0 wt. % AlDr); 5 wt. % AlDr had initial setting time of 140 mins, which also is lower than that of control, and suggesting the presence of AlDr improves the chemical reaction rate of the paste during hydration. The final setting time of 5 wt. % AlDr is 240 mins, but presented a longer setting period and high reaction time than the control.

Table 2: Setting Time of Cement-Al-dross paste

Al-dross (wt. %)	**Initial Setting time (mins)**	**Final Setting time (mins)**	**Setting Period/hardening (mins)**
0	170	240	70
5	140	240	100
10	80	180	100

3.2: Compressive Strength Evaluation of AlDr Mortar Cubes (75x75x75mm)

Curing commences after hydration process (i.e., second day after casting), and under a controlled environment the specimen is expected to maintain the mass concentration as prepared and cast, hence the influence of the curing environment is minimized. The compressive strength test involved fracture failure at ultimate load, which entails breaking (or dissociation) of bond of the concrete cubes and an equivalent to bond strength/energy.

In Table 3, the cubes with 0 wt.% AlDr addition showed highest compressive strength of 8 N/mm^2 on 28^{th} day air curing period. When the cement content of the cube was replaced with 5wt. % AlDr the compressive strength appears stable at 5.4 N/mm^2 after 21-day of curing but increased to 7 N/mm^2 at 28^{th} day curing. The strength characteristics at 10 and 15 wt. % AlDr replacements of cement in the cube do not produce better or comparable strength to the control cube. However, at 20 wt.% AlDr the mix have more pozzolan required for strength build up in the cube and this is the reason for the high strength (8 N/mm^2) on the 28^{th} day of curing, which is similar to that observed for 0 wt.% AlDr concrete cube. This compressive strength can be attributed to the refinement of microstructure, and transformation of CH into CSH gel via

pozzolanic reaction with the occurrence of a dense interface (Yajun and Cahyadi, 2003). It should be noted that aluminum particles in the dross reacts easily with oxygen because of its affinity to form Al_2O_3 and consequently reduce the amount of oxygen required for the strengthening mechanism during curing.

Table 3: Compressive Strength of Cement-AlDr mortar cured in air

Curing (Days)	0 wt. % AlDr N/mm^2	5 wt. % AlDr N/mm^2	10 wt. % AlDr N/mm^2	15 wt. % AlDr N/mm^2	20 wt. % AlDr N/mm^2
7	6.22	5.33	3.45	5.33	4.89
14	6.22	5.78	5.78	4.44	5.78
21	5.33	5.33	3.11	4.44	3.56
28	8	6.67	4.89	4.89	7.56

Table 4 shows the variation of compressive strengths for concrete cubes with partial replacement of cement using AlDr while curing in laboratory water. The 100% cement cube produced maximum strength (8.5 N/mm^2) on the 28th day of curing. For 5 wt. % AlDr there was an apparent strength increase to 7 N/mm^2 on the 21st day but attained stability on the 28th day with a decline in strength (6.8 N/mm^2). The 10 wt.% AlDr cube sample strength achieved stable strength (9.8N/mm^2) onthe28th day of curing. Cubes containing AlDr were observed to develop thin film on the water surface that may retard the cement hydration reaction as it hinders the transportation of oxygen into the material and the blanket film attained it maximum protective covering at 10 wt. % AlDr in the 28th day of curing (Afolabi, 2023). Thus 10 wt.% AlDr showed a better performance than the control cement cube (8.5 N/mm^2). At 15 and 20 wt. % AlDr additions, the cubes' strengths show marginal improvements with an increase in curing days but these are weak compared to control cubes. Thus, the cube strength is enhanced with 10 wt. % AlDr addition.

Table 4: Compressive Strength of Cement-Aldr mortar cured in water

Curing (Days)	0 wt. % AlDr N/mm^2	5 wt. % AlDr N/mm^2	10 wt. % AlDr N/mm^2	15 wt. % AlDr N/mm^2	20 wt. % AlDr N/mm^2
7	5.33	6.22	6.22	6.22	5.33

14	7.11	5.33	4.45	5.33	5.33
21	5.78	7.11	8	6.67	4.89
28	8.45	6.67	9.78	6.67	6.22

In Table 5, the compressive strengths of cubes cured in the University of Lagos lagoon water for 7, 14, 21 and 28th days are presented. The cube strength for the control increases steadily to 21st day (6.8 N/mm^2) before reducing to 4 N/mm^{2}onthe28th day of curing. At 5 and 10 wt.% AlDr additions the cube strengths decreased from 6.3 N/mm^2 at 7th day curing to as low as 1.5 N/mm^2 at 28th day curing. A better performance was obtained when 15wt. % AlDr is used as the strengths increased from 5.8 N/mm^2 on the 7th day to 6.8 N/mm^2 on the 21st day of curing, but drastically reduced on the 28th day (4 N/mm^2). The 20wt. % AlDr addition does not show an improvement in strengths as the cubes decline in strength from the 14th day to the 28th day of curing (5.5-2.5 N/mm^2). In the lagoon water apart from the effect of acidic and corrosive ions, the formation of thin protective blankets owing to the reaction of aluminum metal in the dross with oxygen is rapidly washed off by the ocean currents in the lagoon (Afolabi *et al.,* 2021). On the other hand, aluminum in the dross could also react with chloride ions to form brittle $AlCl_4$.Thus, the bonding activities of the pozzolan are compromised and the cube strengths decline with AlDr addition and with curing days. It should be stated that for most of the test days, the pH of the lagoon water was well above 6.0, that is, $6.0 < pH \leq 7.5$ and for most of these days the environment was acidic with excessive chloride ion > 500 mg/l. The dross is basic in nature and with an acidic environment, an acid-base reaction is quickly initiated and progresses as days of immersion continues resulting in cube material degradation and loss; producing a weak interface between the matrix and the additives.

Table 5: Compressive Strength of Cement-AlDr mortar cured in Unilag lagoon water

Curing (Days)	0 wt. % AlDr N/mm^2	5 wt. % AlDr N/mm^2	10 wt. % AlDr N/mm^2	15wt, % AlDr N/mm^2	20 wt. % AlDr N/mm^2
7	4.45	6.22	6.22	5.78	4.45
14	5.78	4.89	4.89	6.22	5.33
21	6.67	4.00	4.89	6.67	4.89
28	4.45	2.67	1.78	4.00	2.67

3.3: Regression Analysis on Compressive Strengths of Lagoon-Cured Mortar Cubes

The parametric regression equation describing the compressive strength is shown in Equation 1, where a, b, and c are the coefficients of the regression analysis with specific values at different replacement levels of aluminum dross (see Table 6).

Parametric equation: $\sigma(x) = a + bx + cx^2$ ------- (1)

Thus, x the independent variable are the laboratory experimental compressive strength values (N/mm^2) used as regressor variables in the regression model to predict the dependent variable σ (N/mm^2).

The regression model results provide a confident bound of about 95%, which compares adequately with experimental results (see Table 7) indicating the fitness of observation with the parametric results

Table6: Cement-AlDr Mortar Cubes Regression Model Parametric Equations

Al-Dross (wt. %)	Parametric Equations
0	$\sigma(x) = 0.6775 + 0.6467x - 0.0181x^2$
5	$\sigma(x) = 7.33 - 0.1649x + 3.291(10^{-12})\, x^2$
10	$\sigma(x) = 5.55 + 0.1276x - 9.08(10^{-3})\, x^2$
15	$\sigma(x) = 3.002 + 0.4854x - 0.01586x^2$
20	$\sigma(x) = 1.905 + 0.4709x - 0.01582x^2$

Table 7: Cement-AlDr Mortar Cubes Regression Model and Laboratory Experiment Results

Curing (days)	wt.% Al-Dross									
	0		5		10		15		20	
	RM	LE	RM	LE	RM	LE	RM	LE	RM	LE
7	4.32	4.45	6.18	6.22	6	6.22	5.62	5.78	4.43	4.45
14	6.18	5.78	5.24	4.89	5.56	4.89	6.69	6.22	5.4	5.33
21	6.28	6.67	3.87	4	4.23	4.89	6.2	6.67	4.82	4.89
28	4.6	4.45	2.71	2.67	2.01	1.78	4.16	4	2.69	2.67

Conclusion

Concrete specifications and engineering standard describes the curing period as the period required for fresh concrete to become hardened and involves the formation of chemical bonds for solid objects from freshly manufactured plastic concrete and implemented by a controlled curing process to ensure a thermodynamic equilibrium. Partial cement replacement with AlDr is a pozzolanic addition as the cementitious pastes enable binding of the aggregates (though with

reasonably lower strength and bonds). The water-cured specimen gave a satisfactory result with 10 wt. % AlDr addition. This indicates that the cementitious reaction is more active and effective than other AlDr partial replacement levels.

The control provides a maximum strength of 8N/mm^2 with air curing at 28 days, while 10 wt. % AlDr addition presented a maximum compressive strength of 9.78N/mm^2 on 28 days with the water curing method. For all curing days, except on a few instances, there were increments in strength with curing days, with the lowest strength at 7 days and the maximum at 28 days during concrete hardening. This behaviour can be used to predict future concrete strength using as specific parametric equation.

The concrete strength characteristic is asymptotic after 28 days of curing, which enables the consideration of the parametric function of the form: $\sigma(x) = a + bx + cx^2$ for the regression analysis. The regression presented a good correlation that compares with the experimental result within the range of data evaluated with a 95% confidence bound.

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