Research Letter **The Determination of Bound Water in Waste Phosphatic Clay**

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Phosphatic clay is a by-product of phosphate strip mining, particularly in Florida, USA. This waste material occupies about 100 000 acres of land which could be utilized for other causes. Thus, its use as an alternating cementing material for the addition into the mixing matrix of cement paste and concrete to yield higher strength would be profitable for both materials involved. But the biggest drawback faced is that the phosphatic clay possesses high water holding capacity. The water is thus not available for mixing purposes when added to cement paste and concrete and is thus known as bound water. It is therefore essential to determine the amount of bound water to phosphatic clay which shall not be available for the hydration reaction of cement in cement paste and concrete.

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1. Introduction

Since calcium-silicate-hydrate (C–S–H) is primarily responsible for the strength of concrete, it follows that increasing the proportion of C–S–H will result in a stronger material [1]. The addition of a pozzolanic material such as the waste phosphatic clay will induce a reaction with the calcium hydroxide (CH) to convert it into more C–S–H, thereby increasing the density and strength of the concrete structure. Phosphatic clay is a by-product of phosphate strip mining, particularly in Florida, USA. This waste material occupies about 100 000 acres of land which could be utilized for other causes [2]. Thus, its utilization as an alternating cementing material for the addition into the mixing matrix of cement paste and concrete shall produce more C–S–H from the pozzolanic reaction of the CH in the matrix and lead to stronger and denser concrete.

The reaction of cement with water is primarily responsible for the production of hydration products such as C–S–H, CH, and others. Hence the amount of water in a concrete matrix is of utmost importance in the determination of its early stage and time developing properties. The utilization of phosphatic clay comes with a drawback. Waste phosphatic clay has high water holding capacity, and hence it follows that the water that is not present for hydration reactions. The

determination of this bound water is of utmost importance as it would assist in the determination of water available for the hydration of cementitious materials and hence should be determined in order for its utilization in cement paste and concrete for enhanced properties.

2. Background

The waste phosphatic clay for experimentation is attained from PCS Phosphate companies in White Springs, Florida. An elemental analysis of the waste clay from the source is first performed to confirm and determine the extent of silica present for it to react with the CH in the cement matrix to produce more C–S–H and enhance the strength of cement paste and concrete specimens involved [1]. The elemental analysis is performed using a Phillips model PW 2400 X-Ray fluorescence spectrometer.

The biggest drawback faced in the implementation of phosphatic clay waste as an additive to concrete is that the waste material contains a large portion of water, which could be detrimental to the concrete mixture if it is unavailable during the mixing procedure. The clay portion of the waste possesses a very high water holding capacity, and any water held in this way is not available during mixing. This held water will be called bound water and has the capability to alter various physical properties of the clay itself [3, 4]. It is therefore of utmost importance to know the extent of the water bound in this manner, as this water would not be available to increase porosity during mixing and thus should not be considered a part of the water to cementitious materials ratio. On the contrary, this bound water may actually be beneficial since the hydration reaction, once it runs out of readily available water, will tend to pull this bound water away from the clay. The result would be an internal curing mechanism that should lead to better, more extensive hydration of the cement paste matrix within the concrete.

The method used to estimate the amount of bound water is to measure viscosity at various solid concentration levels of the waste clay solution. Excess water beyond the amount that the clay is capable of binding will act as a lubricant, directly affecting the viscosity of the clay-water mixture. As the water content is reduced, a particular solid concentration should be reached where the excess water is no longer present, meaning that its lubricating effect disappears. The result is a steep rise in viscosity as the clay begins to form a solid. This is point where the binding capability of the clay can be distinguished. This trapped water bound by the phosphatic clay should not be a part of the water to cementitious materials ratio.

3. Experimental Approach

There are several ways of measuring the viscosity of solutions. A few techniques are capillary rheometers, the ball drop method, and Brookfield viscometers. The Brookfield viscometer is readily used because of its ease of use and data acquisition. Also, the Brookfield viscometer does not take much time to obtain utilizable data, perhaps in the order of 2 or 3 minutes [5]. On the other hand, a capillary rheometer would take several minutes, and this data would still need to be interpreted using equations that relate the data to the viscosity. For this very reason, the DV I+ Brookfield viscometer is utilized to determine the viscosity of the waste clay solution at various solid concentration levels. Therefore, phosphatic clay from PCS Phosphate at 3% solids content is taken, and the following procedure is implemented for viscosity readings at various solid concentration levels.

- (1) The phosphatic clay from PCS Phosphate is first verified to be at 3% solids content by completely drying a specimen in the oven, weighing the specimen before and after.
- (2) Various specimens of the original waste clay are then dried to various solids concentration levels, with these levels also confirmed through complete drying of a portion.
- (3) The viscosities of the specimens are then measured with the Brookfield viscometer, with all data obtained at 22 degrees C.

TABLE 1: X-Ray fluorescence results for waste clay from PCS Phosphate.

Analyte	Compound formula	Concentration
Al	Al	5.24
Si	Si	22.64
Р	Р	12.22
S	S	0.30
K	Κ	2.21
Ca	Ca	36.18
Ti	Ti	1.40
Fe	Fe	19.50
Sr	Sr	0.30
	Total	100.00

TABLE 2: Viscosities at various solids contents in waste phosphatic clay.

Solids content (wt.%)	Viscosity (cPoise)
3	1600
25	4000
30	4000
35	10200
37	21000
38	68000
39	70000
40	180000

4. Results and Discussion

The elemental analysis of the waste phosphatic clay from PCS Phosphate companies indicates a presence of a silica source to react with CH to produce more C–S–H in the cement matrix for improvements in strengths. Table 1 depicts the results of the elemental analysis.

The DV I+ Brookfield viscometer utilizes hanging spindles that rotate to obtain a viscosity. There are five spindles that accompany this machine, and the viscosity of the solution to be tested will determine which spindle needs to be used. If the wrong spindle is used, the data will not be accurate. Another variable that will affect the viscosity of a solution is the RPM (rotations per minute) of the spindle. This is often used to study polymer-solvent behavior, but in this study we are keeping this constant at 3 RPM.

Table 2 depicts the results for viscosity measurements in centipoises at various solid concentration levels for phosphatic clay. It can be observed from the results that the viscosity begins to rise steeply somewhere around 35% solids. At 40% solids, the clay begins to form a solid, and it can be concluded that there is no longer any water available to act as a lubricant and that it is all bound by the clay. Thus, the amount of bound water for every specimen containing phosphatic clay can be determined by utilizing the fact that at 40% solids none of the water added as part of the waste is available for mixing.

For example, say if we have a mix design with the addition of 100 grams of cement at water to cementitious materials ratio of 0.45, thus, the amount of water required is 45 grams. Now, if a 2% by weight substitution of waste clay is proposed with respect to the amount of cement used, the amount of waste clay becomes 2 grams and the amount of cement is 98 grams. At 40% solid concentration level of waste clay, the rest of the water is unavailable. Therefore, if 2 grams is 40% of a solution, the remaining 60% of trapped water will be 3 grams. Therefore, the amount of water actually present for hydration is 42 grams and not 45 grams. Hence, the new water to cementitious materials ratio is 0.42 and not 0.45. This method of calculation can be followed for all cement paste and concrete specimens prepared with the addition of waste phosphatic clay.

5. Conclusions

From the elemental analysis, it can be concluded that the waste phosphatic clay can serve as a source of silica in the cement matrix and hence has the potential to enhance the compressive and tensile strengths of cement paste and concrete. It hence could be termed as a potential pozzolanic material.

It can also be concluded that the viscosity measurements of the waste phosphatic clay at various solid concentration levels has assisted to determine the extent of bound water in the waste clay. Hence this bound water shall not be considered as a part of the mix design for preparing cement paste and concrete specimens with the addition of waste clay, as it is not available for the hydration reaction of the cementitious materials. The results thus assist in the utilization of phosphatic clay in cement paste and concrete in a more effective manner. Thus the determination of bound water in phosphatic clay has the potential to be profitable for both materials involved and holds the scope to determine the optimal methods for the utilization of waste phosphatic clay in cement paste and concrete in the future.

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