Utilization of Physalis Pith Ash as a Pozzolanic Material in Portland Cement Pastes

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Abstract: Physalis pith ash (PPA) is a renewable and green supplementary cementitious material. It is an effective approach to reduce the quantity of cement that could be used in blended cements. This will reduce the CO₂↑ emission, which in turn will lower the environmental pollution. In the current study, the influence of PPA on physicomechanical characteristics of hydrated cement pastes containing various ratios of PPA (0-30 wt.%) up to 90 days was evaluated. Results showed that the water of consistensy as well as setting times, chemically-bound water content, bulk density and compressive strength increased with PPA content. The apparent porosity and calcium dioxide (or free lime) content were clearly reduced with increasing the PPA content. It is therefore concluded that the cement could be partially replaced by 25 wt.% PPA with no reverse response on the features of cement pastes. Any further increase of PPA content, it affected negatively on all the specific attributes of the hardened cement pastes. So, an optimum PPA content was 25 wt% PPA.

Keywords: Cement, Ash, Consistency, Setting, Free Lime, Porosity, Strength

1. Introduction

1.1. Scope of the Problem

Cementitious composites are the essential building materials for different construction targets regarding to its high strength and durability to improve its performance. In many agricultural countries like Peru, Egypt, Sudan, Tunisia, Morocco, China, India and many others, the gaseous and particulate pollutants from an open burning of agricultural wastes are one of the most common factors of air pollution [1-3]. Physalis is one species of herbaceous plants, growing to 0.4 to 3.0 meters height, which is a plant of the same family of the common Tomato. It is always a stiffer, more upright stem of the Solanaceae family. These can be either annual or perennial. Several plant species of the genus physalis peruviana in the nightshade family Solanaceae, has its origin in Peru. The Physalis and its fruits are commonly called Cape gooseberry and/or golden berry (Figure 1). In Egypt, it is well known as “Harankash”. Egyptians like this fruit to a large extent. Random burning of either fruit covers of Physalis or even any kinds of agro/wastes that generates what is known as “Black Cloud” which pollutes the environment.

The seasonal and highly localized massive burning of these agro-wastes often produced an excessive air pollution that lowers air quality in the surrounding area of Cairo. It is a serious health concern for citizens and authorities [3-5]. The biomaterials power plant, among others, is often exploited for the disposal of bio-wastes like barely and rice husks [5, 6],

Figure 1. Physalis fruit plant.
wheat straw [7], corn stalk [3], sun flower stalk [8] so as to solve the environmental problems. Accordingly, the corn straw (CS) is often incorporating a huge amount of active SiO₂ and Al₂O₃ which helps and has a good pozzolanic reactivity with the released calcium hydroxide resulting from the normal hydration of calcium silicate phases of the cement (CS) is often incorporating a huge amount of active SiO₂ and Al₂O₃ which helps and has a good pozzolanic reactivity with the released calcium hydroxide resulting from the normal hydration of calcium silicate phases of the cement (C₃S and ß-C₂S) as soon as they are becoming in contact with water. So, it can be potentially utilized as an alternative cementitious material to partially substitute by cement [9-16]. Several research studies [17-23] had reported that the use of traditional supplementary cementitious materials could improve and enhance physical, chemical and mechanical features. Recently, the pozzolanic cements are interesting worldwide. These cements are successfully used due to its economical, environmental and technological interests, i.e. decreasing of energy consumption and CO₂↑ emission [22-24].

### 1.2. Targets of the Work

Major targets of the current investigation are to follow the characteristics of Ordinary Portland cement pastes (OPC) containing Physalis fruit cover ash (PCFA) hydrated up to 90 days. The heat of hydration, setting time, water of consistency, bound water and free lime contents, bulk density, apparent porosity and strength development were investigated at all hydration times up to 90 days.

### 2. Experimental and Procedures

#### 2.1. Raw Materials

The raw materials that used in the present study are Ordinary Portland cement (OPC Type I- CEM I 42.5R) with blaine surface area 3400 cm²/g, and Physalis Fruit pith (PP) as a source of active nanosilica with fineness 5155 cm²/g. The blaine surface area was subjected by the “Air Permeability Apparatus”. The OPC sample was provided from Sakkara cement factory, Giza, Egypt, and its commercial name is known as “Asmant El-Montaz”, while PPC sample was supplied by a local market, 6 October City, Egypt. The PPC was first processed and washed with running water, and also with distilled water. Then, it lets to dry under direct sun and open air for several days. The dried PPC was fired at 750 °C inside a suitable oven for one hour soaking time to form Physalis fruit pith ash (PPA). Then, the resulting PPA was sieved using 300 µm standard sieve.

The mineralogical phase analysis of the used OPC sample as determined from Bogue equations [8-12, 25] is shown in Table 1. The chemical constituents of the OPC and PPA as tested by X-ray florescence technique (XRF) is given in Table 2, whilst the mix composition is demonstrated in Table 3. The PPA particles are amorphous and crystalline. It is mainly composed of a large percentage of nano-SiO₂ and a lower percentage of nano-Al₂O₃.

#### 2.2. Preparation and Methods

Seven cement mixtures were prepared from OPC and PPA as 100:0, 95:5, 90:10, 85:15, 80:20, 75:25 and 70:30 having the symbols: P0, P1, P2, P3, P4, P5 and P6, respectively. The various cement batches were blended in a porcelain ball mill using three balls for two hours to assure the complete homogeneity of all batches. The standard water of consistency [12, 25] and setting times [10, 12, 26, 27] of the different cement pastes were measured using Vicat Apparatus. Then, the water of consistency could be determined from equation 1:

$$WC, \% = \frac{A}{C} \times 100$$  \hspace{1cm} (1)

Where, A is the amount of water taken to produce a suitable paste, C is the amount of cement mix (300 g).

During mixing, the exact previously measured w/c-ratio (water of consistency) was poured into the cement portion inside the mixer step by step. The mixer was then run for 5 minutes at an average speed of 10 rpm in order to have perfect homogenous pastes. Before molding of cement cubes, the moulds were covered with a thin film of a motor engine oil to easily the release of the cement cubes from the moulds during the de-moulding process. The cement pastes were then moulded into one inch cubic stainless steel moulds (2.5 x 2.5 x 2.5 cm³) using about 500 g from the cement powder batch, vibrated manually for three minutes, and then on a mechanical vibrator for another three minutes to eliminate all air bubbles tapped inside the cement pastes. The molds were filled to the top surface and smoothed with a flat stainless steel trowel or spatula to obtain a flat and smooth surface [8, 10-12, 14]. After molding
of specimens, the molds must be covered with a wet sheet during the first 24 hours to prevent moisture loss. The molds were then kept in a humidity cabinet for 24 hours under 95 ± 1 relative humidity (RH), and room temperature (23 ± 1°C), demolded in the next day and soon cured by the total immersion in water at an ambient laboratory temperature till the time of testing for heat of hydration, bulk density, apparent porosity, compressive strength, bound water and free lime contents at 1, 3, 7, 28 and 90 days. This is necessary for the cement cubes as it facilitates the normal hydration of cement phases.

The bulk density (BD) and apparent porosity (AP) of the hardened cement pastes [3, 8, 10-12] were determined from the following relations 2 and 3, respectively:

\[ \text{B.D. (g/cm}^3\) = \frac{W1}{W1 - W2} \times 1 \]  
(2)

\[ \text{A.P.\%} = \frac{(W1 - W3)(W1 - W2)\times 100}{W1} \]  
(3)

Where, W1, W2 and W3 are the saturated, suspended and dry weights, respectively. The compressive strength (CS) of the various hardened cement pastes [28, 29] was measured and calculated from the following relation:

\[ \text{CS} = \frac{L (KN)/Sa (cm}^2\) }{(cm}^2\) \times 102 (Kg/cm2)/10.2 (MPa) \]  
(4)

Where, L is the load taken, Sa is the surface area. Thereafter, about 10 grams of the broken specimens were first well ground, dried at 105°C for 24 hours and then were placed in a solution mixture of 1:1 methanol: acetone to stop the hydration [3, 8, 14, 26-29]. The kinetics of hydration in terms of chemically bound water and free lime contents were also measured. About one gram of the sample was first dried at 105°C for 24 hours and then the bound water content was determined on the basis of ignition loss at 1000°C for 30 minutes soaking where about 10 grams of the broken specimens were first well ground, dried at 105°C for 24 hours, and then the chemically combined water content (CWn) at each hydration age was determined on the basis of ignition loss at 1000°C for 30 minutes [29-32] from the following equation:

\[ \text{CWn,\%} = \frac{W1 - W2}{W2} \times 100 \]  
(5)

Where, CWn, W1 and W2 are combined water content, weight of sample before and after ignition, respectively. The free lime content (FLn) of the hydrated samples pre-dried at 105°C for 24 hours was also determined. About 0.5 g sample + 40 ml ethylene glycol → heating to about 20 minutes without boiling. About 1–2 drops of pH indicator were added to the filtrate and then titrated against freshly prepared 0.1N HCl until the pink color disappeared. The 0.1 N HCl was prepared using the following equation:

\[ V1 = N \times 2 \times W \times 100 \times D \times P \times 1000 \]  
(6)

Where, V1 is the volume of HCl concentration, V is the volume required, N is the normality required, W is the equivalent weight, D is the density of HCl concentration and P is the purity (%). The heating and titration were repeated several times until the pink color did not appear on heating. The free lime content [9-12, 14] was calculated from the following relation:

\[ \text{FLn,\%} = \frac{(V\times 0.0033/1)\times 100}{W1} \]  
(7)

Where, FLn and V are the free lime content and the volume of 0.1 N HCl taken on titration, respectively.

3. Results and Discussion

3.1. Chemical Analysis of Cement and PPA

The chemical analysis of PPA in comparison with several pozzolanic materials is indicated in Table 4. According to ASTM C618, 2015a [33], the sum of SiO₂ + Al₂O₃ + Fe₂O₃ requirement for a standard pozzolanic material is 70%, which is more with that of PPA sample (71.91%). The standard specification also sets maximum limits of SO₃ and Loss on Ignition (LOI) as 4% and 10%, respectively. As shown in Table 4, the SO₃ content of PPA sample was found below the acceptable limit, where SO₃ is 0.71% while LOI was negligible (1.23). Therefore, the PPA powder is expected to be as a pozzolanic material in the cementitious system.

**Table 4. Chemical composition of PPA in comparison with CSA, Ghs/S, SF, Pfa, SCBA, SDA, SFSA and WSA.**

| Materials Oxides | PPA | CSA | Ghs/S | SF | Pfa | SCBA | SDA | SFSA | WSA |
|-----------------|-----|-----|-------|----|-----|------|-----|------|-----|
| SiO₂            | 61.61 | 66.47 | 36   | 90.9 | 59.2 | 70.83 | 66.17 | 63.83 | 69.36 |
| Al₂O₃           | 6.93 | 8.29 | 12    | 1.1 | 25.6 | 9.21 | 4.35 | 6.68 | 5.97 |
| Fe₂O₃           | 4.17 | 3.13 | 1     | 1.5 | 2.9  | 1.95 | 2.36 | 2.79 | 2.15 |
| CaO             | 7.65 | 5.46 | 38    | 0.7 | 1.1  | 8.16 | 10.06 | 5.46 | 7.28 |
| MgO             | 3.78 | 4.16 | ----- | 0.8 | 0.3  | 1.32 | 4.41 | 6.23 | 1.44 |
| MnO             | 0.32 | 0.02 | 1.31  | ---- | 1.05 | ---- | 2.19 | ---- | ---- |
| Na₂O            | 3.21 | 2.52 | 0.3   | ---- | 0.2  | 0.12 | 0.08 | 0.24 | 0.31 |
| K₂O             | 1.66 | 2.06 | ----   | ---- | 0.9  | 1.65 | 0.12 | 1.17 | 1.11 |
| SO₃             | 0.76 | 0.51 | 8.11  | 0.4 | 0.3  | 1.47 | 0.30 | 1.11 | 1.27 |
| P₂O₅            | 0.13 | ---- | 2.16  | ---- | 0.14 | ---- | 0.46 | ---- | ---- |
| LOI             | 1.23 | 1.02 | 1.0   | 3.0 | 1.4  | 6.91 | 0.84 | 3.35 | 2.37 |

Abbreviations at the Table (PPA: Physalis ash, CSA: Corn stalk ash, Ghs/S: Granulated blast furnace slag, SF: Silica fume, Pfa: Pulvurizes flay ash, SCBA: Sugarcane bagasse ash, SDA: Saw dust ash, SFSA: Sun flower stalk ash, WSA: wheat stalks ash).
The relationship among the fineness of the OPC (F0) and the different cement batches blended with PFCA (F1-F6) is graphically plotted in Figure 2. As the PPA content enhanced in the cement batch, the fineness of the total mix enhanced too as indicated in Table 3 and Figure 4. It is essentially due to the presence of many nanoparticles of PPA.

3.2. Water of Consistency and Setting Times

Figure 3 illustrates the water of consistency and setting times (initial and final) of the OPC (P0) and the various cement pastes containing PPA (P1-P6). The water of consistency enhanced gradually as the PPA content increased to form suitable cement pastes. This is essentially attributed to the increase in the rate of hydration process in the presence of excess water [25, 34, 35]. Moreover, the PPA ash is divorced for water due to its chemical composition [36, 37]. On the other hand, in spite of the gradual increase of water of consistency, the setting times also increased little by little with PPA content. This is mainly due to the surrounded climatic factors, particularly the very low atmospheric Lab. Temperature (19 °C) during testing [9, 36-39].

3.3. Heat of Hydration

The heat of hydration of the different cement pastes containing PPA (P0-P6) is graphically drawn as a function of cement batches in Figure 4. Results illustrated that as soon as the various cement batch powders become in contact with water, the heat of hydration was soon started to release. The rate of released heat increased as the hydration age proceeded up to 90 days. The same trend was displayed with all cement pastes. This was followed by the release of heat [25, 29]. This is mainly contributed to the increase of the hydration rate of cement pastes as well as the pozzolanic reactions between the ingredients of the cement and those of the PPA. Moreover, the released heat of hydration sharply increased at early ages up to 7 days. This is essentially attributed to the activation effect of the hydration reaction mechanism of $\text{C}_3\text{S}$ by the very fine and active PPA particles [9]. At older ages (28-90 days), the rate of hydration reaction also improved and enhanced leading to the increase of the evolved heat. This may be caused by the activation action mechanism of $\beta\text{-C}_2\text{S}$ with the help of the fine PPA particles [27]. Consequently, the heat of hydration enhanced as the PPA content increased only up to 25 wt% PPA (P1-P5). But with any further increase of PPA content (P6), a sudden sharp decrease of heat of hydration lower than those of the blank (P0) was exhibited. This was contributed to the dilution effect of the main binding material (P0), and the retardation effect of the higher percentage of PPA at the expense of the OPC.

3.4. Chemically-Bound Water Contents

The chemically-bound water contents of the various cement pastes incorporating PPA (P0-P6) are graphically represented versus cement batches in Figure 5. For all cement pastes, the bound water content enhanced and proceeded as the hydration age proceeded up to 90 days. This is essentially
attributed to the hydration of the major cement phases, especially C₃S, C₃A and C₄AF at early ages of hydration up to 7 days, whilst β-C₃S often hydrates at older ages from 28 days onward [25, 26, 29]. The bound water contents enhanced slightly as the PPA content increased only up to 25 wt% (P5), and then suddenly decreased sharply with further increase of PPA nearly at all curing times up to 90 days, i.e. the cement blends P1, P2, P3, P4 and P5 containing 5, 10, 15, 20 and 25 wt% PPA, respectively are slightly above than those of the control (P0). It is essentially attributed to the activation action of the nano- and active silica of the PPA [39-42]. Furthermore, the pozzolanic reactions of PPA through which its ingredients could be reacted with the free lime, Ca(OH)₂ resulting from the hydration of C₃S at early ages of hydration and β-C₂S at older stages to form more CSH. The bound water contents of P6 containing 30 wt% PPA suddenly were decreased sharply to become lower than those of the control (P0). This may be due to that the higher amounts of PPA hindered the hydration of the phases of the blank (P0), and could be acted as an obstacle. Also, the deficiency of the main hydrating material is the true cause for the decrease of bound water [1, 3, 25, 41, 42]. As a result, the optimum replacement of PPA must be only 25 wt% because the higher amounts of PPA is unwanted due to its negative action, i.e. the higher quantity of PPA must be neglected because it may be hindered the hydration of major cement phases.

\[
3C_3S + 6H_2O \rightarrow C_3S_2H_3 + 3Ca(OH)_2 \tag{8}
\]
\[
2\beta-C_2S + 4H_2O \rightarrow C_3S_2H_3 + Ca(OH)_2 \tag{9}
\]

As the PPA content enhanced in the cement mixes (P1-P6), the free lime content decreased nearly at all hydration ages up to 90 days. So, the representing curves of free lime contents of cement batches (P1-P6) was becoming below that of the blank (P0) also at all hydration periods. The increase is attributed to the normal hydration process of the cement phases, while the decrease is due to the activation power of PPA and its pozzolanic reactions between its active nanosilica and nanoalumina with the resulting Ca(OH)₂ from the hydration of C₃S at early times and β–C₃S at older times of hydration in contact of water. In the cement mixes (P1-P5), the rate of hydration improved and enhanced than in the blank (P0) at all hydration stages. This is certainly attributed to activation effect occurred by constituents of PPA, and also its pozzolanic reactivity with the free lime produced by the calcium silicate phases of the cement [41, 42]. The nearly stable values of free lime contents of P6 cement pastes means that the pozzolanic action of PPA stopped completely. This is due to the decrease of the rate of hydration process. In addition to the lack of the essential hydrating material because the higher amount of PPA hinders the rate of hydration [39-42]. The results confirmed that the PPA acts as a pozzolanic material at all ages of hydration. Evidently, the higher amounts of PPA must be neglected due to its negative response on the specific features of the cement [9, 31, 32, 43-45].

3.5. Free Lime Content

Figure 6 shows the free lime contents of the different cement mixes (P0-P6) hydrated up to 90 days were drawn versus cement batches. As a general role, the free lime contents of the control (P0) were continuously increased with the hydration ages up to 90 days showing an improvement in the rate of hydration [13, 25]. This is essentially contributed to the hydration process of major calcium silicate phases (C₃S and β-C₂S) of the cement as follows:

3.6. Bulk Density and Total Porosity

Figures 7 and 8 demonstrate that the bulk density and apparent porosity of the different cement pastes (P0-P6)
were drawn against the cement batches. The bulk density of the various cement mixtures improved and enhanced as the hydration age proceeded up to 90 days, while the apparent porosity decreased. This is principally due to that as the dry cement batches become in contact with water the hydration process begins to produce CSH and/or CAH, which immediately deposited in the pore structure of samples leading to a decrease in the porosity and an increase of bulk density [13, 25], i.e. as the hydration time progresses, the formed hydration products (CSHs) increased too. Therefore, this was reflected positively on both bulk density and total porosity [19, 20, 41]. The bulk density of the cement mixes (P1-P5) increased little by little only up to 25 wt% PPA content (P5), while the total porosity decreased, i.e. the bulk density of cement blends P1-P5 are slightly higher than those of the of the control (P0). This is certainly attributed to the formation of additional CSH and/or CAH due to the pozzolanic reactions of PPA with the constituents of cement through which the constituents of PPA could be reacted with the free lime, Ca (OH)\(_2\) resulting from the hydration of C\(_3\)S at early stages of hydration and β-C\(_2\)S at later stages to produce additional CSH and/or CAH besides those produced from the normal hydration process of cement phases [7, 17-19, 45-48]. With further increase of PPA > 25 wt.% (P6), the BD started to reduce, while the total porosity enhanced at all hydration times. Due to that, the higher amounts of PPA may hinder the hydration process, i.e. it affects adversely and decreases the rate of hydration, but it slightly benefits as a filler [7, 20, 27, 28, 47]. So, the addition of a large amount of PPA at the expense of the main binding material (P0) is undesirable because it was not sufficient for inducing the reaction of cement with nano-active silica of PPA. Hence, it could be concluded that the PPA is so beneficial to cement that it helps in the hydration process as a pozzolanic material and also as a filling agent [7, 21, 28, 45-48].

Figure 7. Bulk density of the various cement pastes (P0-P6) hydrated up to 90 days.

Figure 8. Apparent porosity of the various cement pastes (P0-P6) hydrated up to 90 days.

3.7. Compressive Strength

Figure 9 shows the compressive strength (CS) of the different cement pastes (P0-P6) hydrated up to 90 days versus the cement batches. The compressive strength gradually improved and enhanced as the hydration period proceeded up to 90 days. This is essentially attributed to the formation of CSH and/or CAH that precipitated into the pore structure of the hardened cement samples. This results in a reduction in the pore structure and an improvement in the bulk density. This improved and enhanced the compaction of the prepared samples, besides the good compaction during casting which in turn reflected positively on the compressive strength, As a result, the compressive strength increased [12, 15, 28, 49]. The CS also improved as the PPA content increased at all hydration times only up to 25 wt% PPA (P5). This is essentially due to the activation effect of the active nanosilica and nanoalumina, and moreover due to the formation of more CSH resulting from the pozzolanic reactions of nanosilica and nanoalumina of PPA with the free lime evolved from the hydration of C\(_3\)S and β–C\(_2\)S of cement.

On the other side, the lack of free lime improves the physical, chemical and mechanical properties of the hardened cement pastes. Therefore, the CS improved and enhanced [13, 27, 28, 46-49]. This may be led to the segmentation of large capillary pores and nucleation sites due to the continuous deposition of hydration products (CSH from the normal hydration of cement phases and additional CSH from the pozzolanic reactions of PPA with the released free lime [41, 45, 48, 49]. By further increase of PPA > 25 wt%, the CS values were suddenly decreased sharply. The is mainly contributed to that the replacing of higher amounts of PPA at the expense of the essential cementitious material of the cement stand as an obstacle against the normal hydration process of cement phases. So, the rate of hydration was shortened. This should be reflected negatively on the CS [13, 29-31, 48]. However, the values of CS are still more than those of the blank (P0) at all hydration ages (Figure 9). The
cement mix (P5) recorded the optimum results of CS, whilst that of P6 exhibited the worst. On this basis, the cement batch containing 25% PPA is the optimum cement batch. Hence, the PPA does not only improve the various characteristics of the OPC, but from an economical point of view, it also reduces the cost of the very expensive OPC production.

Figure 9. Compressive strength of the various cement pastes (P0-P6) hydrated up to 90 days.

4. Conclusions

Concerning the findings of the laboratory test results, the following overall conclusions could be obtained:

1. As the nano-silica content from PPA increased in the cement batch, the fineness of the whole batches increased too.
2. The water of consistency of the blank (P0) was 29.55%. The initial and final setting times were 147 and 245 minutes. These values gradually decreased with increasing of PPA content regarding to the pozzolanic reactions of PPA with the silicate phases of the cement, which in turn reduced the mixing water.
3. The heat of hydration, chemically bound water content, bulk density and compressive strength improved and enhanced continuously with the hydration ages. With increasing of PPA content, these properties enhanced up to 25 mass% (P5), and then decreased suddenly with its more increase.
4. The free Ca (OH)₂ content of the blank (P0), and also the various cement pastes (P1-P6) increased with the hydration time. But, with the incorporation of PPA, the free lime contents are becoming lower than those of the blank (P0).
5. On contrast, the total porosity was declined and reduced with the replacing up to 25% PPA (P5), and then increased onward with further increase of > 25 wt% PPA.
6. The improving and enhancing of all properties were achieved by the pozzolanic character of PPA that reduces Ca (OH)₂ coming from the hydration of C₃S and β-C₂S phases of the cement by enhancing the precipitation sites of hydration products. Moreover, the activating and filling effects of the nano silica of the PPA initiated these properties.
7. The replacement of 25 wt% PPA (P5) to Portland cement could be applied with no negative response on the physical, chemical and mechanical characteristics of Portland cement. Hence, it was selected as the optimum mix.
8. In the future, many research trials must be carried out to follow and evaluate the obtained results of the present study.

Compliance with ethical standards

The author declares that there is no conflict of interest anywhere.

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