# Pore Structure Changes of Blended Cement Pastes Containing Fly Ash, Rice Husk Ash, and Palm Oil Fuel Ash Caused by Carbonation

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**Abstract:** In this paper, the effects of carbonation on pore structure of blended pozzolan cement pastes were investigated. Ordinary Portland cement (OPC) was partially replaced with ground palm oil fuel ash (POA), ground rice husk ash (RHA) and classified fine fly ash (FA). The strength, total porosity, specific surface area, and pore size distribution were tested. In general, incorporation of pozzolans increased the total porosity of blended cement pastes in comparison to that of OPC paste. Carbonation of pastes under 5% $CO_2$ and 50% relative humidity (RH) for 28 days resulted in filling of the pore voids and possible attack on calcium silicate hydrate (CSH) depending on the type of pozzolan used. After carbonation, total porosity decreased and specific surface areas of the blended pozzolan cement pastes reduced indicating the infilling of voids. Pore size distributions of POA and RHA pastes were different to those of FA pastes. Large pores of the POA and RHA pastes were increased indicating coarsening of pores owing to possible attack on CSH. It is possible that POA and RHA were highly reactive and hence their uses resulting in severe carbonation compared to use of FA.

## Introduction

It is generally recognized that incorporation of pozzolanic materials as a partial replacement to Portland cement in concrete is an effective means for improving the properties of concrete (Chindaprasirt et al. 2008; Tay 1990). This is due to the fact that calcium hydroxide produced by cement hydration reacts with pozzolan and produces additional calcium silicate hydrate (CSH) gel, blocking existing pores and altering the pore structure. Fly ash (FA) is one of the most common pozzolan and is being used quite extensively. By-products from agricultural process have also been used as pozzolan to improve the properties of cement paste and mortar. These include rice husk ash (RHA) and palm oil fuel ash (POA) (Chindaprasirt and Rukzon 2007).

RHA and POA are available in many parts of the world. The properly burnt RHA at temperature lower than 700°C contains reactive amorphous silica (Della et al. 2002). Rice husk and RHA are still not being fully used owing mainly to their bulkiness and the problems of handling and transportation. For POA, approximately 100,000 tons are produced annually in Thailand (Chindaprasirt et al. 2008). It is a by-product obtained from a small power plant using palm fiber, shells and empty fruit bunches as a fuel and burnt at 800–1000°C. POA is treated as waste and discarded in landfill causing environmental problem.

It is also known that carbonation, a consequence of the transformation of $Ca(OH)_2$ to $CaCO_3$, alters the microstructure of cement paste by decreasing porosity (Ngala and Page 1997). A number of investigations have been directed to study changes in material properties due to carbonation process. Johannesson and Utgenannt (2001) studied the change of specific area of cement mortar using BET theory and reported that specific surface area of carbonated mortar was lower than that of noncarbonated one. The diffusion resistance was adversely affected by carbonation owing to coarsening of pore structures and the effect was more severe with the blended cement paste (Ngala and Page 1997). The effect of carbonation on the concrete is usually enhanced with the incorporation of pozzolans owing to the reduced $Ca(OH)_2$ (Rukzon and Chindaprasirt 2008a). Carbonation can, however, be minimized with the use of low water-to-binder ratio (W/B), low replacement level of pozzolan and increase in curing age in water (Sulapha et al. 2003)

The pore system in well compacted cement-based materials consists of two types of pores (Kumar and Bhattacharjee 2003; Chindaprasirt et al. 2007). The first group consists of gel pores which are micropores with pore radii smaller than 0.01 μm and are included in the volume occupied by CSH. The second group consists of capillary pores which are mesopores with average radii ranging from 0.01–10 μm. The volume of capillary pores and distribution of air voids affect strength and permeability properties of cement-based materials. The smallest gel pores associated with the CSH phase, however, have significant influence on creep and shrinkage.

Mercury intrusion porosimetry (MIP) is a common technique used for characterizing the pore structure of cement paste. The technique consists of submerging a sample in mercury, applying pressure in step, and recording the volumetric changes after each step. Assuming cylindrical pores, the Washburn equation is used to relate applied pressure to pore size (Washburn 1921). The transport properties of porous materials are highly dependent on

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**Table 1.** Physical Properties and Oxides of OPC, FA, and POA

| Physical properties and oxides | OPC | FA | POA | RHA |
|---|---|---|---|---|
| Median particle size ($\mu$m) | 15.0 | 4.9 | 7.2 | 10.2 |
| Retained on a sieve No. 325 (%) | — | 1–3 | 1–3 | 1–3 |
| Specific gravity | 3.14 | 2.45 | 2.25 | 2.23 |
| Blaine fineness ($cm^2/gm$) | 3,600 | 5,700 | 11,800 | 11,200 |
| $SiO_2$ | 20.9 | 41.1 | 63.6 | 93.2 |
| $Al_2O_3$ | 4.8 | 21.6 | 1.6 | 0.4 |
| $Fe_2O_3$ | 3.4 | 11.3 | 1.4 | 0.1 |
| CaO | 65.4 | 14.4 | 7.6 | 1.1 |
| MgO | 1.3 | 3.3 | 3.9 | 0.1 |
| $Na_2O$ | 0.2 | 1.1 | 0.1 | 0.1 |
| $K_2O$ | 0.4 | 2.6 | 6.9 | 1.3 |
| $SO_3$ | 2.7 | 2.2 | 0.2 | 0.9 |
| LOI | 0.9 | 2.5 | 9.6 | 3.7 |
| $SiO_2+Al_2O_3+Fe_2O_3$ | — | 74.0 | 66.6 | 93.7 |

the volume fraction of porosity, surface area of pores, and connectivity of the pore network.

In this study, ordinary Portland cement (OPC), FA, ground POA and ground RHA are used as pozzolanic materials to study the effect of carbonation on pore structure of blended cement paste. MIP is used to assess changes of pore parameters in the blended cement pastes. Total porosity, changes in pore sizes, and surface area were determined.

## Materials and Experiment Details

### Materials

OPC type I, lignite FA from Mae Moh power plant in the northern part of Thailand, POA from a thermal power plant in the south of Thailand, RHA from open burning at maximum temperature of 670°C, and type-*F* superplasticizer (SP) were the materials used. Fine FA also with 3% retained on a sieve No. 325 was obtained from air classification of as-received coarse FA. Ground POA and ground RHA were obtained using ball mill grinding until the percentages retained on a sieve No. 325 were 3%. The physical properties and chemical compositions of OPC, FA, RHA, and POA are given in Table 1. The Blaine fineness of the OPC, FA, POA, and RHA were 3,600, 5,700, 11,800 and 11200 $cm^2/g$, respectively. The specific gravity of OPC, FA, POA, and RHA were 3.14, 2.45, 2.25, and 2.23, respectively. Their particle size distributions are given in Fig. 1.
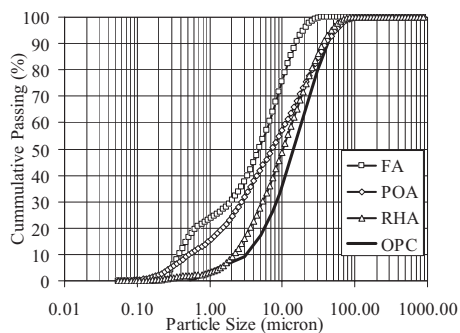


**Fig. 1.** Particle-size distribution of FA, POA, RHA, and OPC

The loss on ignition of POA was rather high at 9.6%. Cook and Suwanvitaya (1981), however, reported a finding that the loss of ignition of RHA around 10% is not detrimental to the strength development of the blended cement. The as-received POA was, therefore, used as it had also been shown to be suitable for use as pozzolan (Chindaprasirt et al. 2008).

### Mix Proportions and Curing

Pozzolans were used to replace OPC at dosages of 0%, 20%, and 40% by weight of binder. The W/B of 0.30 was used for all mixes shown in Table 2. SP was incorporated to obtain suitable consistency of paste.

### Compressive Strength Test

Cube specimens of 50 mm were used for compressive strength test. Specimens were cast in two layers with tamping of 32 tamps per layer [as specified in ASTM C109 (2001)] followed by 10 s of vibration at the end. The cast specimens were covered with polyurethane sheet and damp cloth in a 23±2°C chamber. They were demolded at the age of 1 day and kept in water at 23±2°C until testing age. Compressive strength testing was done at ages of 7, 28, and 90 days. The reported results are an average of three samples.

### Carbonation and Mercury Intrusion Porosimetry (MIP) Tests

For carbonation and MIP tests, samples were prepared as described in section 2.3 for 50 mm cubes. At the age of 28 days,

**Table 2.** Paste Mix Proportions

| Mix | OPC | FA | POA | RHA | SP (%) |
|---|---|---|---|---|---|
| OPC | 100 | — | — | — | 1.8 |
| 20FA | 80 | 20 | — | — | 0.4 |
| 40FA | 60 | 40 | — | — | 0.2 |
| 20POA | 80 | — | 20 | — | 1.8 |
| 40POA | 60 | — | 40 | — | 3.0 |
| 20RHA | 80 | — | — | 20 | 2.0 |
| 40RHA | 60 | — | — | 40 | 3.4 |

Note: SP (%) by weight of binder.
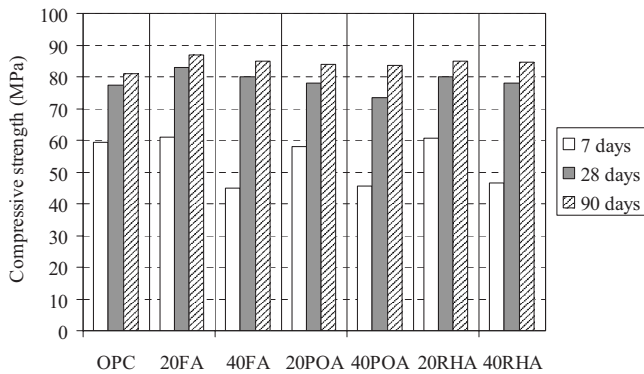
**Fig. 2.** Compressive strength of pastes at 7, 28, and 90 days



**Fig. 3.** Total porosity of pastes (noncarbonated and carbonated)

four discs of 3 mm thickness were cut from the middle section of cylinders. Two discs were stored hermetically (noncarbonated) at 50% relative humidity (RH) and $23 \pm 2°C$, while another two discs were stored in 5% $CO_2$ chamber (carbonated) maintained under same RH and temperature conditions. At the age of 56 days, these samples were split and their middle portions were used for MIP test. The samples for MIP were frozen at $-195°C$ by immersion in liquid nitrogen for 5 min followed by evacuation at a pressure of 0.5 Pa at $-40°C$ for 48 h as suggested by other researchers (Kumar and Bhattacharjee 2003; Galle, 2001; Konecny and Naqui 1993). Measurements on total porosity, capillary porosity, gel porosity, pore size distribution and specific surface area were then carried out using a MIP with pressure range from 0 to 30,000 psi capable of measuring pore size diameter down to 0.007 μm.

## Results and Discussions

### Compressive Strength

The results of compressive strength of pastes are given in Fig. 2. The 7, 28, and 90-day strengths of OPC paste were 59.5, 77.5, and 81.0 MPa. At the age of 7 days, the compressive strengths of cement pastes similar for 20% dosage of pozzolan but decreased significantly with an increase in the replacement of pozzolans to 40% dosage. At the age of 28 days, the strengths of pastes containing pozzolans were approximately the same as that of OPC paste. At 90 days, the strengths of pastes containing pozzolans were slightly higher than that of OPC paste by up to 6 MPa. This later age strength development is characteristic of pozzolanic materials. By comparing among FA, POA, and RHA pastes, the compressive strengths of FA pastes were slightly higher than those of POA and RHA pastes at the same age. FA consists largely of spherical shape particles and its presence in cement mixtures improves workability due to ball-bearing effect, improves the dispersion of cement particles, and increases packing density of the solid material (Payá et al. 1996; Isaia et al. 2003) Better dispersion of cement grains and formation of more gel phases largely account for the strengthening effect (Xu and Sarkar 1994). In this experiment, classified FA was quite fine with Blaine fineness of 5700 $cm^2/g$ and hence its filling effect could be high. The incorporation of RHA and POA may also have produced a filler effect due to its fine particle size (Chindaprasirt et al. 2008). The ground RHA contains very irregular-shaped particles with a porous cellular surface. (Chindaprasirt and Rukzon 2007). The POA consists mainly of irregular-shaped particles (Chindaprasirt et al. 2008).
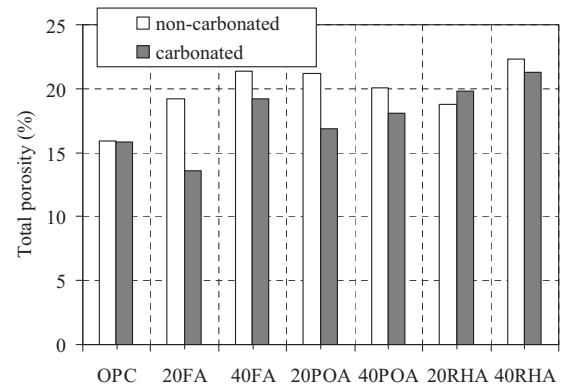
The dispersing effect of POA and RHA on the cement particles has not been reported and their additions to cement mixtures reduce the workability. However, their reactivity was high due to their high surface areas. Comparing between RHA and POA paste, their compressive strengths were similar. RHA pastes showed marginally higher strength than POA pastes at the same age owing probably to the lower loss of ignition (LOI) of RHA of 3.7% in comparison to 9.6% of POA.

### Pore Structure

**Total Porosity**

Fig. 3 shows results of total porosity of the carbonated and noncarbonated pastes obtained by MIP. For noncarbonated pastes, the total porosity of blended cement pastes increased with the incorporation of pozzolans and with the increase in the dosage levels as compared to that of normal cement paste. Total porosity of OPC paste was 16.0%. For FA, POA, and RHA pastes, total porosities increased to 19.5% and 22.0%; 21.5% and 20.0%; 18.5% and 22.5% for 20% and 40% dosages, respectively. This is consistent with reports in literature that total porosity of the cement paste increases as a result of the incorporation of pozzolans (Chindaprasirt and Rukzon 2007; Neville 1995; Chindaprasirt et al. 2005). Poon et al. (1997) also found increase in porosity of pastes with partial replacement of cement with FA and suggested that this might be due to the higher W/C ratio and lower gel/space ratio when cement was partially replaced.

After carbonation (carbonated samples), total porosities of pastes were generally reduced in comparison to those of the noncarbonated pastes as shown in Fig. 3. These results also indicate that the reductions were related to the type of pozzolans used. FA pastes produced the largest reductions of 3.0%–6.0% followed by POA pastes and RHA pastes of 2.0%–4.5% and −1.5%–1.5%, respectively. The reduction in the pore volume is due to deposition of $CaCO_3$ formed during the carbonation (Ngala and Page 1997). The deposition of $CaCO_3$ is in turn related to the availability of the $Ca(OH)_2$ in the cement pastes. It can be expressed that pastes containing RHA had the smallest amount of $Ca(OH)_2$ owing to high reactivity and high fineness of RHA (Chindaprasirt et al. 2008; Rukzon and Chindaprasirt 2008b). This results in a smaller amount of $CaCO_3$ formed during carbonation thus the RHA pastes showed smallest reduction in porosity compared to FA and POA pastes. Reductions in porosity of the pastes containing POA were slightly higher than those of RHA pastes. The Blaine fineness of POA (11,800 $cm^2/g$) was similar to that of RHA (11,200 $cm^2/g$). However, the $SiO_2$ content of POA was

**Table 3.** Summary of Threshold Pore Diameters of Noncarbonated and Carbonated Pastes

| Mix | Threshold pore diameters ($\mu$m) | |
| --- | --- | --- |
| | Noncarbonated | Carbonated |
| OPC | 0.0468 | 0.0390 |
| 20FA | 0.0489 | 0.0428 |
| 40FA | 0.0523 | 0.0468 |
| 20POA | 0.0432 | 0.0495 |
| 40POA | 0.0363 | 0.0601 |
| 20RHA | 0.0352 | 0.0393 |
| 40RHA | 0.0318 | 0.0328 |

lower and the LOI was higher than those of RHA. This would result in a higher amount of the $CaCO_3$ in POA pastes as compared to those of RHA pastes. In addition to a different elemental composition, the Blaine fineness of FA was much less than the fineness levels of RHA and POA. This could have resulted in larger formation of $CaCO_3$ in the FA pastes compared to RHA and POA pastes. It may be pointed out here that an advanced stage of carbonation can result in increase of porosity owing to the formation of silica gel from $CO_2$ attack on CSH (Neville 1995). It is of interest to note that total porosity of carbonated cement paste was marginally lower than noncarbonated cement paste. Normal cement paste contained a full supply of $Ca(OH)_2$, the porosity of carbonated cement paste should be lower. The results suggest that there could be other mechanisms involved in carbonation and further investigation is needed to verify this.

**Pore-Size Distribution**

Results of the pore size distributions determined using MIP are summarized in Table 3 and examples of which are shown in Fig. 4. The curve peak of 20FA.N (noncarbonated) and 20FA.C (carbonated) pastes were located at 0.0489 and 0.0428 $\mu$m, respectively. While peaks of capillary pores of 40FA.N (noncarbonated) and 40FA.C (carbonated) pastes were located at 0.0523 and 0.0468 $\mu$m. These results suggest that pore diameters of OPC and FA pastes decreased as a result of carbonation. But threshold pore diameters of POA and RHA pastes (carbonated paste) increased after 28 days of carbonation regardless of their dosage levels. For example, threshold pore diameters for 20POA.N and 20POA.C pastes were 0.0432 and 0.0495 $\mu$m and for 20RHA.N and 20RHA.C pastes the diameters were 0.0352 and 0.0393 $\mu$m, respectively. These results indicated that pore structures of POA

and RHA carbonated pastes were coarser in comparison to that of FA pastes. When $Ca(OH)_2$ has been depleted, CSH is attacked and silica gel formed resulting in coarser pore systems (Neville 1995; Zhang et al. 2004). Since POA and RHA were very fine and of high $SiO_2$ content, they were more likely to consume high amount of $Ca(OH)_2$ compared to FA. Rapid depletion of $Ca(OH)_2$ in POA and RHA pastes could have accelerated carbonation attack on CSH increasing their capillary porosities and pore diameter. The results thus suggest that FA was more effective in resisting carbonation than POA and RHA.

**Classification of Pore Size**

The volumes of mercury intrusion in a specified range of pore were normalized with the total volume of mercury intrusion for each paste. To determine the distribution of pores, the normalized volumes of mercury intrusion with various ranges of pore sizes of 0.007–0.01, 0.01–0.05, 0.05–0.1, 0.1–1.0, 1.0–10, and 10.0–100.0 $\mu$m were calculated. The results in Fig. 5 show the differences in the structures of FA, POA, and RHA pastes. For the noncarbonated pastes, FA pastes showed greater volumes of large 10.0–100.0 $\mu$m pores than the OPC paste, whereas the POA and RHA paste showed lower volumes at this range of pores. The small 0.007–0.01 $\mu$m pores of the FA pastes with 40% FA were slightly larger than for OPC paste, but POA and RHA pastes showed significantly higher volumes at this range of small pores. These results agree with postulation that POA and RHA were more reactive than FA. It may be pointed out here that though, POA and RHA had high silica contents, their burning temperature and thus reactivities were different. In addition, POA and RHA were ground to very high fineness levels and hence filler effect could partly be responsible for the increase in small pores.

For the carbonated pastes, the small 0.007–0.01 $\mu$m pores of all pastes were reduced especially for the POA and RHA pastes in comparison to those of the noncarbonated pastes. Amount of these small pores were similar to that of OPC paste. Amount of the large 10.0–100.0 $\mu$m pores in the FA pastes reduced in comparison to those of the noncarbonated FA pastes and carbonated OPC paste. However, the proportion of their pore sizes increased in POA and RHA carbonated pastes in comparison to their noncarbonated pores. Amount of these large pores of POA pastes were slightly reduced and those of RHA paste were increased compared to carbonated OPC paste. It is suggested that carbonation was severe in POA and RHA pastes as they contained smaller amount of $Ca(OH)_2$ possibly owing to their high reactiv-
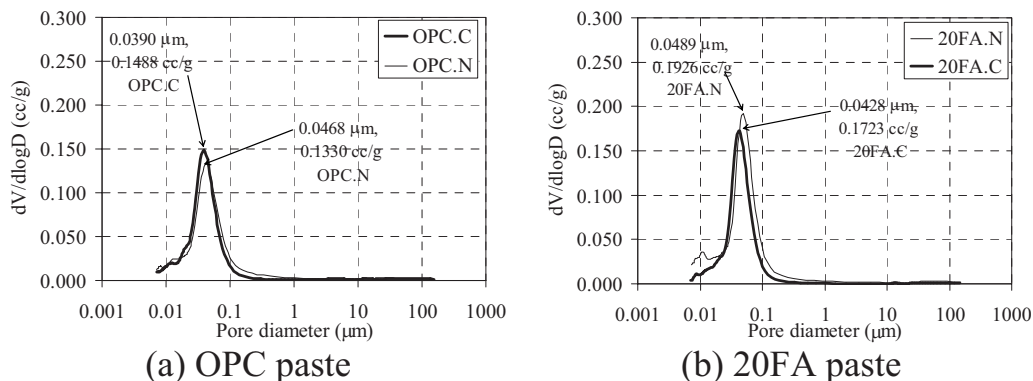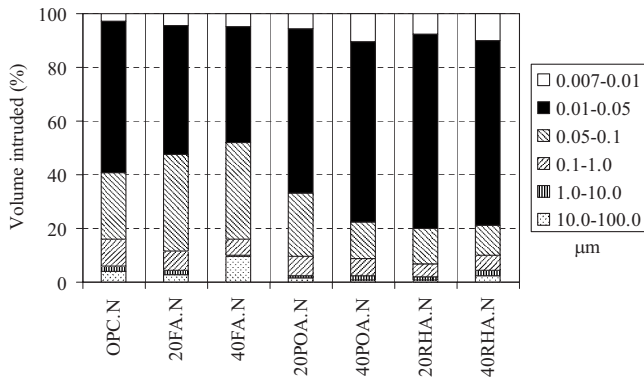


(a) OPC paste
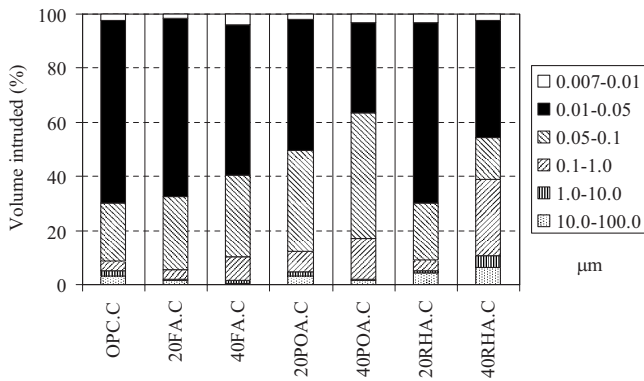


(b) 20FA paste

**Fig. 4.** Example of pore size distribution of cement pastes ($N$=noncarbonated and $C$=carbonated)

**(a)** non–carbonated



**(b)** carbonated

**Fig. 5.** Classification of pore sizes of pastes (noncarbonated and carbonated)

ity relative to FA. The increases in large pores along with corresponding decrease in small pores indicate attack on CSH in the POA and RHA carbonated pastes.

**Specific Surface Area**

The changes of specific surface area with and without carbonation paste are presented in Table 4. The effect of carbonation on the specific surface area of OPC paste was negligible. The specific surface areas of carbonated pozzolan pastes, however, were consistently lower than those of the noncarbonated ones. Area reductions in carbonated FA pastes were smaller than reductions for

**Table 4.** Values of Specific Surface Area for Carbonated and Noncarbonated Pastes

| Mix | Specific surface area ($m^2/g$) | | Difference | |
|---|---|---|---|---|
| | Noncarbonated | Carbonated | ($m^2/g$) | (%) |
| OPC | 8.33 | 8.50 | 0.17 | 0.01 |
| 20FA | 10.82 | 8.04 | −2.78 | −0.30 |
| 40FA | 12.20 | 11.46 | −0.74 | −0.09 |
| 20POA | 22.78 | 8.68 | −14.12 | −3.22 |
| 40POA | 20.51 | 8.79 | −13.71 | −3.08 |
| 20RHA | 17.63 | 14.38 | −3.25 | −0.57 |
| 40RHA | 27.81 | 11.81 | −16.00 | −4.45 |

POA and RHA pastes. These findings were consistence with results of the pore size distribution. Decreases in specific surface area indicate reduction in of smallest pores in the system owing to crystallization of calcite crystals in the smallest pores and/or attack on CSH.

## Conclusions

From tests done, the results show that incorporation of FA, POA, and FA pozzolans led to increase in total porosity of blended cement pastes relative to OPC paste. The carbonation of pastes causes infilling of pore voids and the possible attack on CSH at the advanced stage of carbonation. Total porosity, specific surface area and pore size distribution were variably affected by carbonation and the type of pozzolans used. Results clearly showed that total porosity of carbonated pastes was lower due to deposition of $CaCO_3$ in pores. Reductions in specific surface were smaller in carbonated FA pastes than in POA and RHA. The volumes of large $10.0–100.0$ $\mu$m pores in carbonated FA pastes reduced in comparison to corresponding noncarbonated pastes, but increased for carbonated POA and RHA pastes suggesting that carbonation effect was severe in POA and RHA pastes in comparison to FA pastes. The increases in large pores along with corresponding decrease in small pores indicate attack on CSH in the POA and RHA carbonated pastes.

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