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EFFECTS OF SUPERABSORBENT POLYMER ON MECHANICAL PROPERTIES AND POROSITY OF CEMENT-BASED MATERIALS

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ABSTRACT: Superabsorbent polymer (SAP) is a powdered chemical mixture that can be used for internal curing during the hardening of cementitious materials, helping to prevent fine cracks that may arise. In addition to its role in internal curing, SAP can affect the workability and hydration process of the materials. As SAP has only been studied in recent years, there is no standard theory or methodology for its utilization. In this study, various parameters were evaluated to determine the mechanical properties influenced by the use of superabsorbent polymer (SAP), including the size of the SAP particles, the condition of the SAP during mixing, curing conditions, and the types of supplementary cementitious materials used. These variations were assessed based on the compressive strength of the specimens at 28 days. Subsequently, X-Ray μCT was used to identify the effect of SAP on pore structure. The results indicated that specimens incorporating 0.2% SAP/binder and supplementary cementitious materials exhibited a compressive strength that was 22.6% higher than specimens using only supplementary cementitious materials. This finding demonstrates that SAP can significantly enhance the compressive strength of cement-based materials by facilitating internal curing and increasing the degree of hydration. By improving the properties of cementitious materials, the use of SAP allows for a reduction in the amount of cement required, contributing to more sustainable construction practices.

Keywords: Superabsorbent Polymer, Mortar, Pore Structure, Internal Curing, X-Ray μCT.

1. INTRODUCTION

Mortar is a composite material composed of cement, sand, water, and various additives. In certain circumstances, supplementary cementitious materials (SCMs) such as fly ash or silica fume can be used to replace a portion of the cement. This substitution offers multiple advantages: fly ash contributes to environmental sustainability by reducing industrial waste, is cost-effective, enhances compressive strength, and generates less heat during hydration. However, the hydration process for fly ash is slower than that of cement, resulting in a prolonged hydration period. This slower hydration rate also leads to lower heat generation and helps in preventing fine cracks [1]. On the other hand, silica fume substitution has a positive impact at an early age by improving the bond between the aggregate and matrix, resulting in a less porous structure [2].

A ternary cement system can also be employed to overcome the limitations of individual supplementary cementitious materials (SCMs). For example, fly ash and silica fume are often used in combination, with silica fume enhancing early-age properties and fly ash contributing more significantly at later ages.

Effective hydration relies heavily on maintaining the relative humidity within the mortar, making the curing mechanism critically important. Proper curing ensures that the mortar retains its moisture content, allowing hydration to proceed without interruption. The most common curing method is external curing, which is straightforward, such as water curing and natural curing. Water curing involves immersing the test specimen in water, while natural curing is achieved by exposing the specimen to open air. However, internal curing has potention to be more effective in ensuring continuous hydration. One promising internal curing medium currently under study is the superabsorbent polymer (SAP).

As an internal curing agent, SAP can maintain relative humidity [3] by supplying water up to 2-3 mm around its pores [4], thus sustaining the hydration process and reducing self-desiccation. This mechanism is expected to improve mechanical properties, such as compressive strength. However, using SAP also has some drawbacks, such as reduced workability and a potential decrease in compressive strength due to increased macro-porosity from SAP voids. When using 0.2-0.5% SAP, the decrease in compressive strength can range from 10-20% for SAP particles of 300-500 μm, and from 30-50% for particles of 50-150 μm [4].

Pores play a crucial role in understanding the effect of SAP in cement-based materials, making it essential to conduct a detailed analysis of pore structure and pore size distribution. Traditional tools such as mercury intrusion porosimetry (MIP) [5, 6] and scanning electron microscopy (SEM) [7] utilize indirect methods and come with notable limitations, often resulting in inaccurate or misleading outcomes. In contrast, X-ray μCT serves as a direct method for examining the internal structure of cement-based materials. As a cutting-edge technology, it provides advanced capabilities for analyzing and visualizing the internal pore structure [8, 9].

Despite these challenges, SAP as an internal curing medium can further optimize the use of SCM, ensuring all cementitious particles are adequately hydrated. Achieving perfect hydration of cement enhances mechanical properties while ensuring no cement is wasted. This optimization reduces cement consumption, lowers costs, and contributes to environmental sustainability.

This article is organized into five sections. The first section introduces the fundamental theory utilized in this study. The second section highlights the significance of the research and its contributions. The third section outlines the experimental methodology employed. The fourth section presents the results and provides a comprehensive discussion. Finally, the fifth section concludes with the key findings of the study.

2. RESEARCH SIGNIFICANCE

This research aims to evaluate the role of SAP in binary and ternary mortar phases with various SAP parameters, including particle size, mixing conditions, and curing methods. Additionally, this study will verify changes in pore structure through X-Ray µCT analysis.

This analysis demonstrates the significant impact of SAP on compressive strength and pore structure, highlighting the importance of mixing conditions and the potential benefits of internal curing.

3. EXPERIMENTAL METHODOLOGY

3.1 Materials

Table 1. Composition of cementitious materials (%)

	Cement	Fly Ash	Silica Fume
SiO_2	20.4	47.7	96.4
Fe_2O_3	9.84	6.14	0.108
Al_2O_3	6.40	24.7	0.551
SO_3	2.44	0.755	1.24
CaO	55.4	10.5	0.459

The oxide compositions of cement, fly ash, and silica fume are shown in Table 1. The fine aggregate used is Galunggung sand, with its physical properties detailed in Table 2 and its aggregate gradation depicted in Fig. 1. The superplasticizer used is of the polycarboxylate type, which enhances workability. The particle sizes of the SAP used are presented in Table 3 and visualization in Fig. 2.

The difference in particle size of SAP was studied to determine which size is most suitable for internal curing medium. If the particle size is too small, the water absorption may be insufficient for effective internal curing. Conversely, if the particle size is too large, it may result in larger macropores, which can negatively impact the mechanical properties.

Table 2. Physical properties of Galunggung sand

Bulk Specific Gravity (Dry)	2.509
Bulk Specific Gravity (SSD)	2.555
Apparent Specific Gravity	2.629
Water Absorption	1.81%
Moisture Content	4.47%
Clay Lumps	5.26%
Compacted Bulk Density	1.74
Loose Bulk Density	1.48
Fineness Modulus	2.6

Table 3. Particle size of superabsorbent polymer

SAP	Particle Size (µm)
Large	1000
Small	250

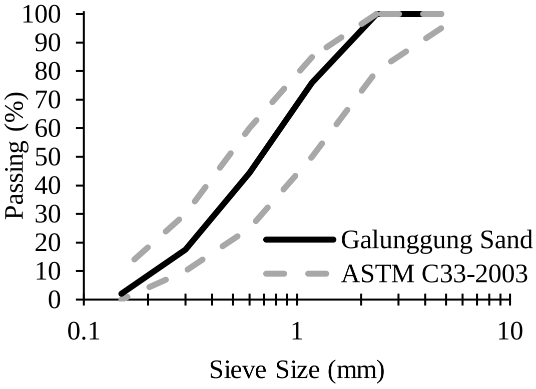


Fig. 1 Particle size distribution of fine aggregate

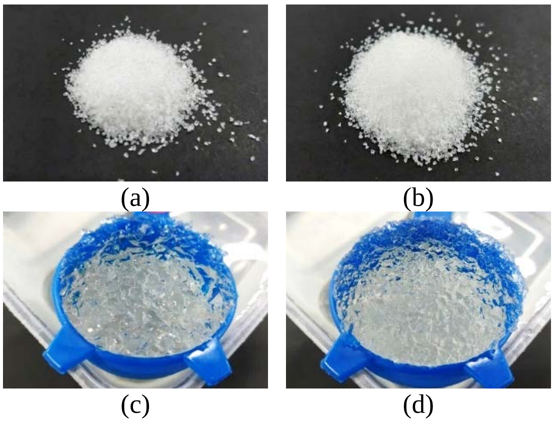


Fig. 2 SAP: (a) Large-dry; (b) Small-dry; (c) Large-presoaked; (d) Small-presoaked

3.2 Mixture Proportion

Table 4 shows the mix design of the mortar based on trial mixing. To limit the scope, a fixed SAP content of 0.2% was used. Previous studies have examined various SAP contents and found that 0.2% is the optimum value for achieving internal curing, thereby enhancing mechanical properties such as compressive strength [6].

Specimen notation is based on the abbreviation of the size and condition of the SAP during mixing; for example, large SAP mixed in a dry state is abbreviated as LD. The last letter identifies the curing conditions: N for natural curing and W for water curing. The subsequent term describes the amount and type of supplementary cementitious materials used. Binary specimens replace 20% of the cement with a supplementary cementitious material, while ternary specimens use two supplementary cementitious materials, each replacing 20% of the cement. A 20% replacement of cement with SCM was used based on a study that found the optimum fly ash content to be 20% for achieving optimal compressive strength at 28 days[10]. This replacement level is suitable for this study, which also evaluates compressive strength at 28 days.

The mixing procedure is carried out as follows:

a. Weigh the materials according to the mix design.
b. Add cement, sand, SCM, and SAP (if dry). Mix at 48 RPM for 1 minute.
c. Add water and superplasticizer to the mixture. Mix at 48 RPM for 3 minutes.
d. Add SAP (if presoaked). Stir at 48 RPM for 1 minute.
e. Stir at 88 RPM for 4 minutes and 144 RPM for 2 minutes.

3.3 SAP Absorption and Desorption

The absorption and release capacity of SAP were measured using the tea bag method and the filtration method, as outlined in the RILEM study [11]. These two methods were employed to compare their effectiveness.

3.4 Rheology

Rheology of fresh mortar properties were tested with flow table test by its spread diameter according to ASTM C1437.

3.5 Compressive Strength

The compressive strength test was carried out according to ASTM C39 at 28 days with nine spesimens 5×5×5 cm for each batch. The age test of the mortar was limited to 28 days, as the primary focus of the study was to examine SAP's effectiveness as an internal curing medium. Each concrete will be removed from its mold after 24 hours and cured with natural or water curing until the age of tests.

3.6 Imaging Methods

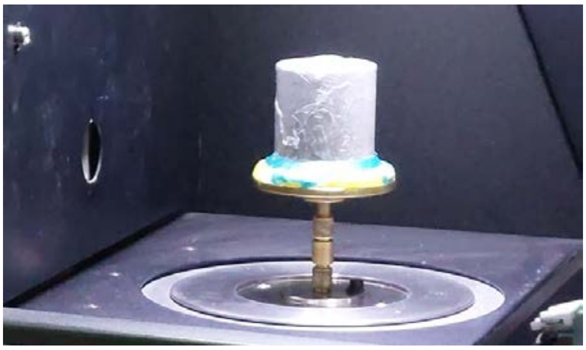


Fig. 3 Scanning X-Ray μCT

The X-Ray μCT imaging system utilized in this research was used SkyScan 1173, show in Fig. 3, to evaluate porosity, pore thickness, and pore structure of the specimens. ImageJ used to processing the images and using Plugin BoneJ [12–14] to obtain thickness of pore.

Table 4. Mix design (kg/m^3)

SAP	Cement System	OPC	SCM 1	SCM 2	Fine Aggregate	SP	SAP	Mixing Water	Presoaked Water
Without SAP	Non	667	-	-	1333	13.3	-	200	-
	Binary	533	133	-					
	Ternary	400	133	133					
Dry SAP	Non	667	-	-	1333	13.3	1.3	200	-
	Binary	533	133	-					
	Ternary	400	133	133					
Presoaked SAP	Non	667	-	-	1333	13.3	1.3	186.67	13.3
	Binary	533	133	-					
	Ternary	400	133	133					

4. RESULTS AND DISCUSSIONS

4.1 SAP Absorption and Desorption

Table 5. Behavior of SAP

SAP	Water Absorption (g/g)	Water Desorption (%)
Large	266	5.72
Small	201	4.71

Comparison between the two absorption methods for large SAP in water is shown in **Fig. 4(a)**. Initially, the test was conducted using the tea bag method with 0.2 g of SAP. However, the absorption of SAP was limited because the tea bags constrained the SAP. The test was then performed using the filtration method with 0.8 g of SAP to obtain more accurate results. Nevertheless, the top portion of the SAP was not directly exposed to water during swelling, limiting its absorption capacity. Subsequently, the filtration method was adjusted to use 0.4 g of SAP. It is essential for the filtration method to ensure that all SAP particles are in direct contact with water and that the amount of SAP tested is suitable for the size of the strainer used.

Comparison between the two absorption methods for small SAP in water is shown in Fig. 4(b). The tea bag method produced results similar to those observed with large SAP, where the tea bags constrained the SAP, allowing it to absorb only about 80% of its capacity. Based on the experience with large SAP, the filtration method was employed using 0.3 g and 0.4 g of SAP. The results indicated that both 0.3 g and 0.4 g of SAP produced similar outcomes, demonstrating that the strainer used is adequate for 0.4 g of SAP.

Desorption was tested using the filtration method to allow direct water release. The test results are presented in Table 5.

4.2 Rheology

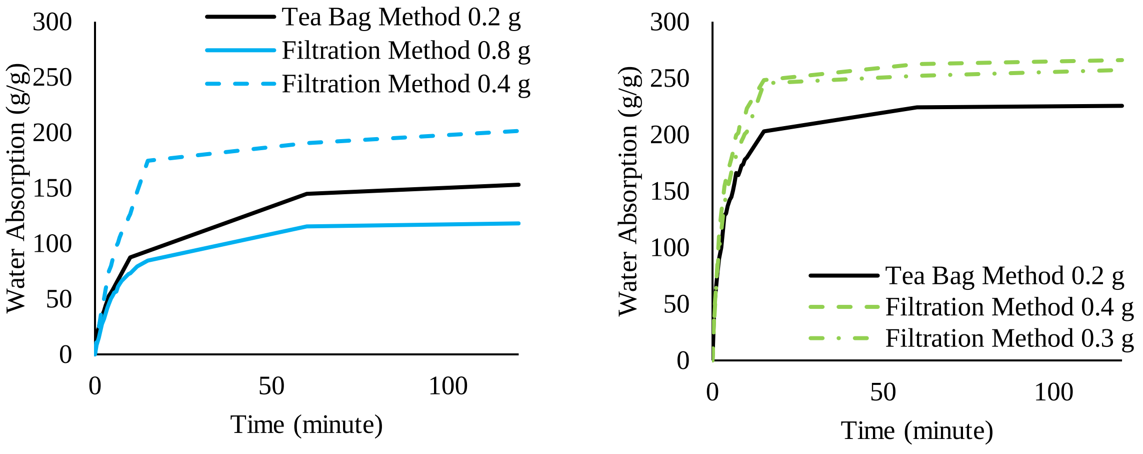
Fig. 5(a) shows specimens using SCM without SAP, which illustrates that the specimen with fly ash replacement exhibited increased workability due to the low carbon content. Conversely, the specimen with silica fume replacement showed decreased workability due to the smaller particle size compared to cement. Using silica fume as a substitution increases the total surface area, resulting in more water being absorbed, which decreases the amount of free water available. This effect of silica fume replacement on water absorption is also observed in a previous study [15].

Fig. 5(b) shows specimens using SAP without SAP. The influence of SAP reduces workability because some of the mixing water is absorbed, so it seems the w/c has decreased. The workability of using SAP in dry and presoaked conditions is lower than reference specimen.

4.3 Compressive Strength

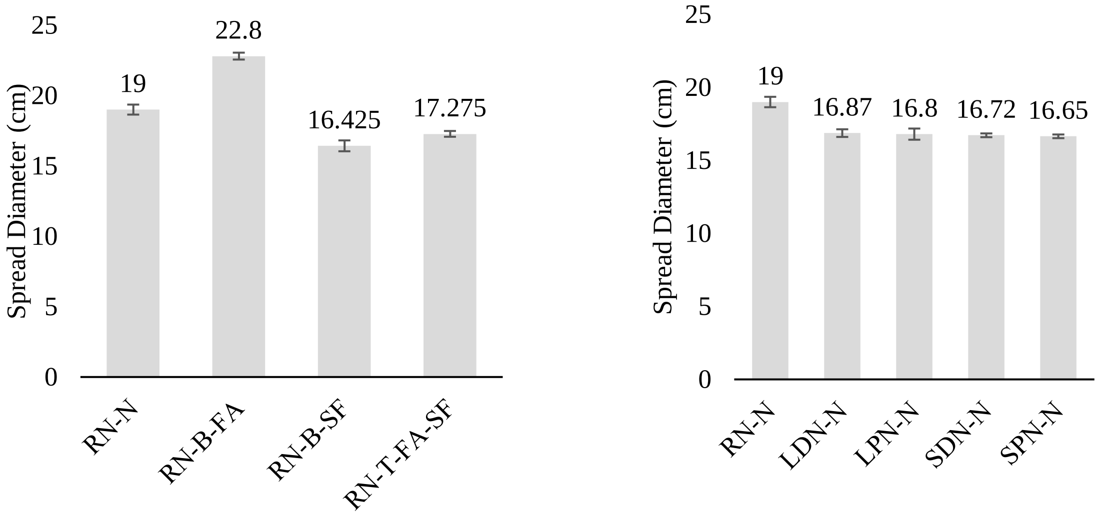
Table 6 shows compressive strength of all specimens using natural and water curing, visualized in Fig. 6.

First, specimens incorporating SAP exhibit a reduction in compressive strength, as observed from specimens labeled 'N' in both natural and water curing. Theoretically, this reduction is due to the pores formed by the SAP particles. Larger SAP sizes further decrease the compressive strength. For example, the small-dry specimen exhibits a 16.6% lower compressive strength compared to the reference specimen in natural curing. This result aligns with the RILEM report, which found a reduction in compressive strength of 10-20% for SAP particles of 300-500 μm [4].



(a) (b)

Fig. 4 Tea bag method and filtration method on water absorbtion: (a) Large SAP; (b) Small SAP

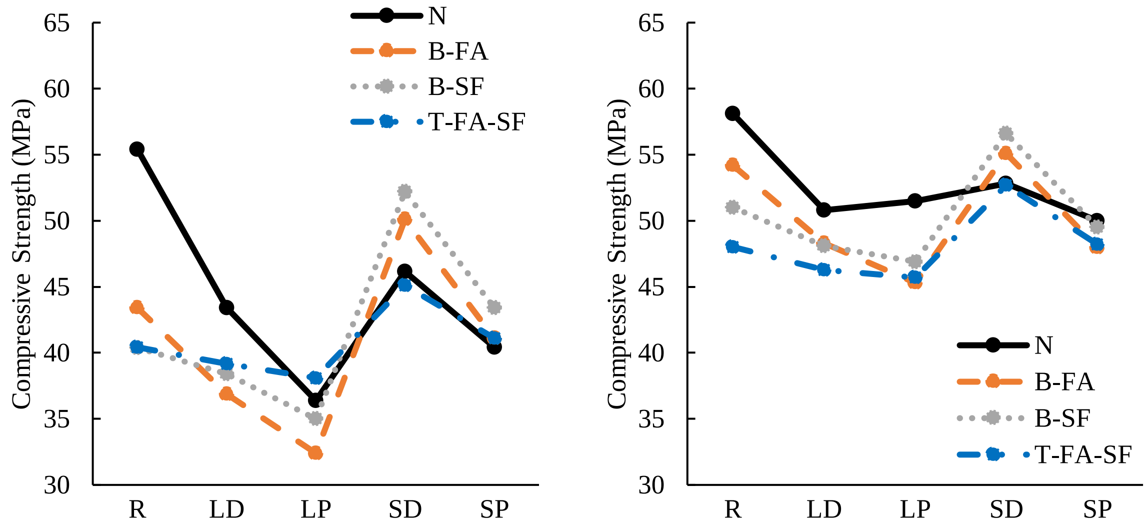


(a) (b)

Fig. 5 Effect on spread diameter flow table test: (a) Variation of supplementary cementitious materials; (b) Variation of size SAP and mixing conditions

Table 6. Compressive strengths of natural and water curing (MPa)

Particle Size-Mixing Condition of SAP	Natural Curing				Water Curing			
		Binary		Ternary		Binary		Ternary
	Normal	Fly Ash	Silica Fume	Fly Ash & Silica Fume	Normal	Fly Ash	Silica Fume	Fly Ash & Silica Fume
Reference	55.4	43.4	40.4	40.4	58.1	54.2	51.0	48.0
Large-Dry	43.4	36.9	38.4	39.2	50.8	48.3	48.1	46.3
Large-Presoaked	36.4	32.4	35.0	38.1	51.5	45.3	46.9	45.7
Small-Dry	46.2	50.1	52.2	45.1	52.8	55.1	56.6	52.7
Small-Presoaked	40.4	41.1	43.4	41.1	50.0	48.0	49.5	48.2



(a) (b)

Fig. 6 Effect curing method on compressive strength: (a) Natural curing; (b) Water curing

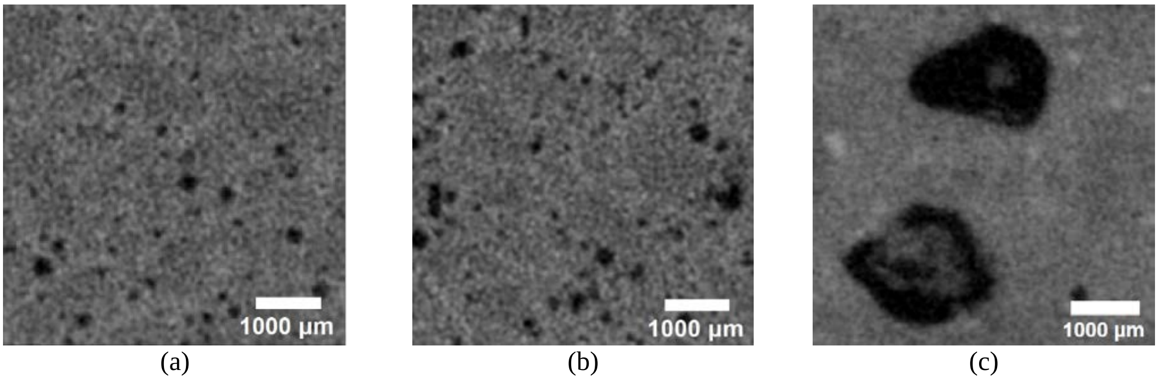


Fig. 7 2D Slice image: (a) Non; (b) Dry-SAP; (c) Presoaked-SAP

Second, specimens using SCM have lower compressive strength compared to those not using SCM. This observation is derived from specimen 'R' in both natural and water curing. The lower strength results from the hydration process of SCM requires more than 28 days to achieve complete hydration compared to ordinary Portland cement (OPC) [1, 16].

Third, using SAP with supplementary cementitious materials (SCM) produces different results. For example, the small-dry silica fume specimen exhibits a 22.6% higher compressive strength than the reference silica fume specimen in water curing. SCM hydration continues after 28 days, potentially surpassing the reference specimen. The increase in compressive strength can be attributed to the secondary hydration mechanism facilitated by the internal curing of SAP, which maintains relative humidity and leads to a higher degree of hydration. Furthermore, the small-dry fly ash specimen with natural curing (50.1 MPa) were able to almost same with water curing (54.2 MPa). This observation can be explained by a study in [17], which suggested that $Ca(OH)_2$ forms from soluble calcium ions in SAP. $Ca(OH)_2$ continues to form until it occupies half the volume of the SAP pores. This additional $Ca(OH)_2$ can act as a catalyst for secondary hydration with SiO_2 or Al_2O_3 from SCM, resulting in the production of CSH or CAH.

Fourth, small SAP particles enhance hydration more effectively than large SAP particles. With the same weight, small SAP particles have a greater number of particles, leading to more uniform distribution within the specimen. For example, the small-dry specimen shows a 6.4% higher compressive strength than the large-dry specimen in natural curing. Therefore, internal curing is more effective because a larger area is covered by small SAP particles. In addition, larger SAP particles result in larger macropores forming after the SAP releases water, leading to increased porosity and pore thickness. Higher porosity can adversely affect the mechanical properties [18] and durability [19].

Fifth, among all test specimens using supplementary cementitious materials, small-dry specimens exhibit higher compressive strength than small-presoaked specimens. This is because the water absorbed during mixing is not as substantial in the presoaked condition, resulting in smaller pores.

Overall, the use of SAP without SCM leads to a reduction in compressive strength due to the formation of macropores. Similarly, the use of SCM without SAP results in reduced compressive strength at early ages due to slower hydration processes. This study found that SAP can enhance compressive strength by increasing the degree of hydration. Additionally, there is significant potential for cost savings through reduced cement usage. When combined with SCM, these effects are amplified, offering additional cost-saving potential from cement replacement and contributing to more sustainable construction materials practices.

4.4 Pore Structure

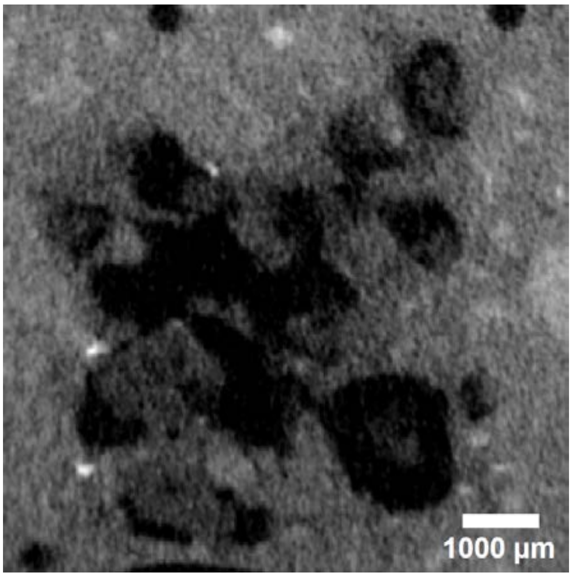


Fig. 8 SAP Sticking Each Other

Fig. 7 shows 2D slice image for specimens without SAP (Non), using SAP in dry conditions

(Dry-SAP), and using SAP in presoaked conditions (Presoaked-SAP). Visually, Fig. 7(b) appears identical to the conventional mortar in Fig. 7(a), but has more pores. Meanwhile, Fig. 7(c) shows the presence of macropores due to the expanded SAP that can absorb free water directly.

On the other hand, Fig. 8 shows how SAP affects the pore structure when the particles are not well separated from each other, leading to the formation of porous areas. This is particularly evident in the presoaked condition, where it becomes difficult to separate the SAP particles.

Table 7. Pore properties

Pore Properties	Non	Dry-SAP	Presoaked-SAP
Porosity	7.64%	9.06%	10.56%
Thickness (μm)	173	187	494

Table 7 shows the pore properties presented as porosity and thickness. The porosity result of the Non specimen (7.64%) using the imaging method is quite reliable when compared to a previous study (7.43%) [20]. It can be seen that the use of dry-SAP increases porosity due to the SAP becoming a pore after releasing water. However, the thickness of dry-SAP remains relatively the same as the non specimen suggesting that using Dry-SAP does not significantly affect the pore structure. On the other hand, presoaked-SAP, which causes macropores, significantly increases thickness, almost tripling it. The macropores of SAP observed through 2D slice images explain the phenomenon of decreased compressive strength due to become a weakness area.

Pore structure can be observed from pore thickness distribution, shown in Fig. 9. Dry-SAP has a peak thickness similar to Non, but with more number of pores at 300 μm. This is compensated by a smaller number of pores at thicknesses <200 μm and >400 μm due to internal curing. On the other hand, Presoaked-SAP has a right-shifted distribution with a peak at 400 μm, so thickness pore more larger and cause reduction of mechanical properties and durability.

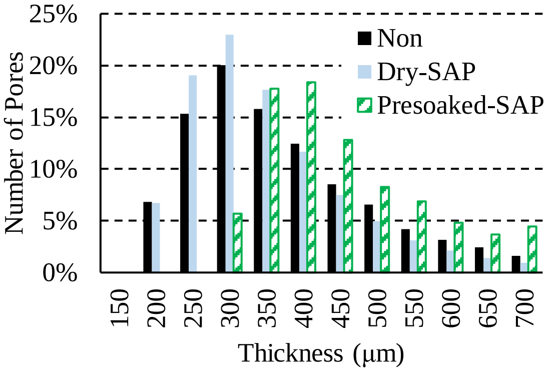


Fig. 9 Pore thickness distribution

5. CONCLUSIONS

Based on the study, it can be concluded that:

1. The filtration method is the most suitable for determining the absorption and desorption capacity of SAP, as the tea bag method only allows for approximately 80% of SAP's capacity to be utilized.
2. SAP particles with a size of 250 μm result in 6.4% higher compressive strength than those with a size of 1000 μm, as larger SAP particles have the potential to become macropores and create porous areas due to sticking together.
3. Based on mechanical properties and pore structure, it can be concluded that SAP is suitable as an internal curing medium, particularly when using SCM and small particles of SAP, which enhance compressive strength by up to 22.6%.
4. Using SAP in combination with SCM offers potential cost savings through reduced cement usage or replacement and also promotes more sustainable construction materials.

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