Mechanical strengths and ultrasonic pulse velocity evaluation of supersulfated cement mortar containing sodium sulfate

Herry Suryadi, Jean Jessica, Djayaprabha Pangestu, Tiffany Candra

Abstract. The study was conducted to evaluate the mechanical strengths and ultrasonic pulse velocity in the presence of sodium sulfate (SSC). Sodium sulfate was employed as a supplementary cementitious material with various amounts (up to 25 wt.%) of GGBFS. The results exhibited that the inclusion of Na\textsubscript{2}SO\textsubscript{4} could improve the 28\textsuperscript{th} day compressive strength by about 2.94 to 4.28. However, the results showed that the inclusion of Na\textsubscript{2}SO\textsubscript{4} also increased the ultrasonic pulse velocity (UPV) value. The 28\textsuperscript{th} day UPV value for the mixture without the addition of Na\textsubscript{2}SO\textsubscript{4} was 2931 m/s. The results also showed that the inclusion of Na\textsubscript{2}SO\textsubscript{4} significantly improved the 28\textsuperscript{th} day flexural strength by about 2.5 times higher when compared with the mixture without Na\textsubscript{2}SO\textsubscript{4}. Additionally, the addition of Na\textsubscript{2}SO\textsubscript{4} also showed a positive effect on the increase of the splitting tensile strength. The results showed a significant improvement of splitting tensile strength when compared with the mixture without Na\textsubscript{2}SO\textsubscript{4}. The inclusion of Na\textsubscript{2}SO\textsubscript{4} also resulted in a significant increase of compressive strength and ultrasonic pulse velocity (UPV).

1 Introduction

The utilization of Portland cement as a binding material in the production of mortars and concrete has been regarded as a major role in the construction industry. Portland cement production still uses fossil fuels and consumes a tremendous amount of energy, contributing around 2-5% of total global energy consumption. Furthermore, a huge amount of carbon dioxide (CO\textsubscript{2}) was released into the atmosphere as a result of the limestone decomposition process[1] employed for manufacturing cement as well as materials transportation[2]. Approximately one ton of CO\textsubscript{2} was released for every ton of cement manufactured[3]. Due to the environmentally harmful process of producing Portland cement, several researchers focused on the utilization of solid industrial waste such as ground granulated blast furnace slag (GGBFS), an industrial waste by-product obtained from the smelting of iron ore, in blast furnaces to produce iron or steel products[4]. GGBFS has been frequently used as a supplementary cementitious material that could be advantageous for cement substitution in the production of slag blended cement[5] or alkali-activated slag with various alkali activators[6-7].

The supersulfated cement (SSC), as an environmentally friendly cement, can serve as an alternative to Portland cement due to its being mainly composed of ground granulated blast furnace slag (GGBFS) in the range of 70 to 90%, a sulfate activator, such as gypsum, in the 10 to 20% range, and an alkaline activator, such as Portland cement clinker, in the amount of 1 to 5%[8]. The SSC contained sulfur trioxide (SO\textsubscript{3}), which was the primary component in sulfate-activating GGBFS, in a higher amount (up to 25 wt.% of gypsum), when compared to the SO\textsubscript{3} content in the slag blended cement, which only has an amount of SO\textsubscript{3} of approximately 3 wt.%[5]. Furthermore, the small amount of Portland cement added as an alkali activator in SSC could raise the pH, provide calcium hydroxide, and promote the dissolution of GGBFS. The aluminum, calcium, and silicon from dissolved GGBFS are then reacted with gypsum to produce ettringite and calcium silicate hydrate (C-S-H) gels[9]. The most important advantage of SSC was its high resistance to sulfate attack and low-heat hydration[10]. However, SSC's low early strength gain has been a significant limitation[11].

On the other hand, sodium sulfate (Na\textsubscript{2}SO\textsubscript{4}), which is widely available in nature, could act as a sulfate activator for GGBFS[12-13]. In Na\textsubscript{2}SO\textsubscript{4} activation of GGBFS provided C-S-H gels, in a low Ca/Si ratio, with a significant aluminium substitution known as C-A-S-H, which contributed to increase the strength development. Additionally, ettringite was also developed in the subsequent reaction[14].

There is still limited study on utilizing Na\textsubscript{2}SO\textsubscript{4} as a source of sulfate in SCC for developing sustainable materials. Therefore, the effect of Na\textsubscript{2}SO\textsubscript{4} substitution of GGBFS in SSC was investigated. In this study, SSC was utilized as the binding agents of supersulfated cement mortar (SSM) that were composed from GGBFS that was activated with Na\textsubscript{2}SO\textsubscript{4} as a sulfate activator, with the SO\textsubscript{3} content varied at the amount of 0 wt.%, 2.5 wt.%, 7.5 wt.%, and 10 wt.% and ordinary Portland cement (OPC), as an alkaline activator, at a fixed amount of 5 wt.%. Studies were carried out into the
mechanical strengths of SSM, including its compressive strength and flexural strength, as well as its ultrasonic pulse velocity (UPV) evaluation. The OPC-based mortar (OPCM) was also prepared as the reference mortar specimens, and the findings were compared with them.

2 Experimental Methods

2.1 Materials

The SSC utilized in this research was composed of three kinds of powders. They are GGBFS produced by PT. KRNG Indonesia, OPC manufactured by PT. Semen Indonesia, Tbk., and Na₂SO₄ obtained from PT. Bratachem. The densities of powdered materials were investigated by using the La Chatelier flask according to ASTM C188 [15]. The densities of GGBFS, OPC, and Na₂SO₄ were 2.85 g/cm³, 3.05 g/cm³, and 2.66 g/cm³, respectively. The fine aggregate (hereinafter called sand) that was utilized to produce mortar was obtained from Galunggung Mountain, West Java, Indonesia, with a specific gravity of 2.57 and an absorption capacity of 2.22% that was investigated by using ASTM C128 [16]. The sand had a fineness modulus of 2.55 and a gradation curve that satisfied the ASTM C33 [17] limits, as shown in Fig. 1.

![Gradation curve of sand](image)

**Fig. 1.** Gradation curve of sand.

Dynamon NRG 1030 superplasticizer (SP), supplied by PT. Mapei Indonesia Construction Solutions, with a density of 1.04±0.2 g/cm³ at 20°C, was utilized for controlling the expected flowability of the fresh mortar.

2.2 Mixture Proportions

The SSM that developed in this study was composed of GGBFS, OPC, and Na₂SO₄ as binder ingredients. The mixtures of SSM were proportioned by using the volumetric method with the fixed amount of OPC at 5 wt.%. The amount of Na₂SO₄ was varied to achieve the SO₃ amounts of 0 wt.%, 2.5 wt.%, 7.5 wt.%, and 10 wt.% as a partial replacement of GGBFS by mass. The sand was added to the mixture along with one part of binder to 2.5 parts of sand. The water-binder ratio (w/b) was fixed at 0.4. The amount of SP, that proportioned to binder was determined based on the trial mix results. In addition, OPCM, as the reference mortar specimen, was produced with the equivalent w/b as comparison. The mixture proportions of SSM and OPCM could be found in Table 1.

<table>
<thead>
<tr>
<th>Mixture Code</th>
<th>Quantity (kg/m³)</th>
<th>SP (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SSM0</td>
<td>235 558 0 29 1470</td>
<td>0.3</td>
</tr>
<tr>
<td>SSM2.5</td>
<td>235 532 26 29 1470</td>
<td>2.0</td>
</tr>
<tr>
<td>SSM7.5</td>
<td>235 480 78 29 1470</td>
<td>1.0</td>
</tr>
<tr>
<td>SSM10</td>
<td>235 454 104 29 1470</td>
<td>0</td>
</tr>
<tr>
<td>OPCM</td>
<td>235 – – 0 1470</td>
<td>0.3</td>
</tr>
</tbody>
</table>

**Table 1.** Mixture proportions.

SSM = supersulfated cement mortar, the numbers denote the SO₃ amount, OPCM = OPC-based mortar, W = water, G = ground granulated blast furnace slag, Na₂SO₄ = natrium sulfate, OPC = ordinary Portland cement, S = sand, SP = superplasticizer

2.3 Mixing and Casting Process

The SSM mixing process was started by placing powder (e.g. GGBFS, OPC, and Na₂SO₄ for making SSM or OPC for making OPCM) together with sand into the mixer and stirring in a dry state for 3 minutes to ensure that all materials were homogeneously mixed. As the mixer continues running, poured the mixing water into the mixer and stirred for 3 minutes. The mixer was stopped for a while and the SSM mixture was stirred manually to ensure the mixture well mixed. Then, the exact amount of SP was added to the mixture and the mixer was run for 2 minutes. Soon after completing the mixing process, the fresh mortar was tested for its flowability (will be discussed in Subsection 3.1). After the expected flowability was obtained, the fresh SSM was poured into molds and the fresh SSM was compacted with the tamper. One day after the molding process, hardened SSM specimens were released from the mold, treated using the sealed curing method, and stored in plastic containers at laboratory ambient temperature of about 23 ± 3°C with a relative humidity (RH) above 50% until the day of testing.

2.4 Specimens and Testing Standards

In this study, the cube and prism specimens used were prepared in triplicate for each test. The 50-mm cube specimens were made for compressive strength determination according to ASTM C109 [18] by using a compression testing machine (ELE model ADR2000), as shown in Fig. 2. The loading on the specimen was applied in the range of 900 to 1800 N/s and the peak load (Pₘₐₓ) from the compression test was recorded. The SCM compressive strength (fₘ) was computed using Equation 1 by dividing Pₘₐₓ by the actual loaded surface area (A).

\[ fₘ = \frac{Pₘₐₓ}{A} \]
Fig. 2. Compressive strength testing.

The prism specimens, with a size of 40×40×160 mm, were made for performing the center-point flexural strength test according to ASTM C348 [19] by using a universal testing machine (Hung Ta model HT 9501), as demonstrated in Fig. 3. Prior to testing, the actual width (b) and height (d) of the specimen were measured by a digital calliper, and then the specimen was placed with a support span (L) of 120 mm. The load was applied at a rate of 2640±110 N, the maximum load was recorded (P) and the flexural strength (f_r) could be calculated by using Equation 2.

\[ f_r = \frac{3PL}{2bd^2} \] (2)

Fig. 3. Flexural strength testing.

The prism specimens, with a size of 50×50×200 mm, were prepared for conducting UPV evaluations, a well-known non-destructive test, which was tested by using a portable UPV device (Proceq model Pundit Lab+) by adopting ASTM C597 [20] a testing standard for concrete. A pair of 54 kHz transducers were utilized for transmitting and receiving compressional ultrasonic waves. Prior to testing, the actual specimen’s length (L) was measured using digital calliper and the portable UPV device calculated the travel time (t) of the compressional waves. The UPV value (V_{UPV}) could be calculated by using Equation 3. The UPV evaluation on mortar specimen was demonstrated in Fig. 4.

\[ V_{UPV} = \frac{L}{t} \] (3)

Fig. 4. UPV testing.

In addition, the UPV value at 28 days could possibly be used to determine the quality of hardened mortar. Estévez et al. [21] described the cement mortar quality assessment based on UPV values, as shown in Table 2.

Table 2. Mortar quality based on UPV value [21].

<table>
<thead>
<tr>
<th>V_{UPV} (m/s)</th>
<th>Mortar Quality</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt; 3800</td>
<td>Excellent</td>
</tr>
<tr>
<td>3800-3500</td>
<td>Good</td>
</tr>
<tr>
<td>3500-3200</td>
<td>Poor</td>
</tr>
<tr>
<td>&lt; 3200</td>
<td>Very Poor</td>
</tr>
</tbody>
</table>

3 Results and Discussions

3.1 Flowability Test

The flowability test was performed on every fresh SSM mixture according to ASTM C1437 [22]. The flow percentage of the fresh mortar was calculated by comparing the increase in the average final flow diameter (D_{avg}), after 25 drops using flow table apparatus, conforming to ASTM C230/C230M [23] to the initial diameter. Table 3 showed the results of the flowability test. The flow percentage ranged from 100% to 108%, which indicated all mortar had a similar workability that was controlled by SP.

Table 3. SSM and OPCM flowability.

<table>
<thead>
<tr>
<th>Mixture Code</th>
<th>D_{avg} (mm)</th>
<th>Flow (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SSM0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SSM2.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SSM7.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SSM10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>OPCM</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
3.2 Compressive Strength

The development of compressive strength for six different mortar mixtures was illustrated in Fig. 5. It was noticeable that the compressive strength of the SSM mixture could be increased by adding Na$_2$SO$_4$. The compressive strength of SSM without any Na$_2$SO$_4$ incorporation (SSM0), only reached 4.61, 6.27, 8.48, and 10.50 MPa at the ages of 7, 14, 28, and 56 days, respectively. When compared to SSM0, the addition of Na$_2$SO$_4$ with the SO$_3$ amounts of 2.5 wt.% (SSM 2.5), 7.5 wt.% (SSM7.5), and 10 wt.% (SSM10) increased the 28-day compressive strength by approximately 2.94, 4.06, and 4.26 times, respectively. It was obvious that OPCM has a higher compressive strength than SSM. When compared to OPCM, the compressive strengths of SSM0, SSM2.5, SSM7.5, and SSM10 were lower by 82.91, 49.81, 30.52, and 27.27%, respectively. At the age of 56 days, the compressive strength of SSM mixture with Na$_2$SO$_4$ addition could still increase by 21.1, 21.3, and 18.0% for SSM2.5, SSM7.5, and SSM10, respectively, when compared with the compressive strength at the age of 28 days. At the later age, the SSM with Na$_2$SO$_4$ addition of 7.5 wt.% (SSM7.5) and 10 wt.% (SSM10) continuously gained its compressive strength increment, so that was quite comparable with OPCM. That could be investigated by the compressive strength development at 56 days of SSM7.5 and SSM10, which lowered only about 12.81 and 12.42% when compared to OPCM. As discussed previously, the increment of compressive strength mainly contributed by the formation of ettringite and C-S-H gels from the hydration process that occurred in the SSM.

![Fig. 5. Compressive strength test results.](image)

3.3 Flexural Strength

The ability of the mortar specimens to resist bending from the applied load could be determined by their flexural strength. The results of the flexural strength of hardened mortar specimens that were conducted in this study can be found in Fig. 6. Based on those findings, it could be seen that the flexural strengths of hardened mortar increased during the curing process, which was associated with the continuous process of GGBFS activation with Na$_2$SO$_4$ and the development of the hydration products [24]. This was consistent with the compressive strength results. However, certain marginal fluctuations in the flexural strength test results found in this study were probably caused by an inhomogeneous mortar matrix. The SSM0, SSM2.5, SSM7.5, and SSM10 had 28-day flexural strengths of 1.96, 5.66, 7.41, and 6.39 MPa, respectively. After 28 days, the OPCM mixture had a flexural strength of 7.67 MPa. When compared with the OPCM mixture, it could be found that the mixture with the incorporation of Na$_2$SO$_4$ in the SO$_3$ amount of 7.5 wt.% had an equivalent flexural strength that was approximately 3.38% lower, while the mixture of the SO$_3$ amount of 10 wt.% had a lower flexural strength by about 13.96%.

![Fig. 6. Flexural strength test results.](image)

3.4 Ultrasonic Pulse Velocity

The results of UPV evaluation of the mortar specimens were demonstrated in Fig. 7. The addition of Na$_2$SO$_4$ to the SSM mixture significantly raised the UPV values due to the hydration process and the inclusion of a sulfate activator which could refine the microstructure [25]. At the age of 28 days, the mixture of SSM0, SSM2.5, SSM5, and SSM10 had UPV values of 2931, 3687, 3942, and 3940 m/s, respectively. Based on those results, the quality of mortar could be classified as described in Table 2. The mixture without any Na$_2$SO$_4$ addition only could be classified as very poor quality. The addition Na$_2$SO$_4$ as little as 2.5 wt.% (SSM2.5) could improve the quality to good mortar. Furthermore, the addition of Na$_2$SO$_4$ at 7.5 wt.% (SSM 7.5) and 10 wt.% (SSM10) could enhanced the quality of mortar to excellent grade. When compared with OPCM, the UPV value of SSM2.5, SSM7.5, and SSM10 was lower by 11.42, 5.32, and 5.28%, respectively. In general, the increment of UPV value due to the formation hydration product [26] which is corresponding with its mechanical strengths improvement.
The correlation of mortar compressive strength \( (f_m) \), and UPV value (\( V_{UPV} \)) was frequently proposed by the exponential equation [27-29] with \( a \) and \( b \) as empirical coefficients, which might be stated in Equation 4.

\[
f_m = ae^{bV_{UPV}}
\]  

(4)

The relationship between compressive strength and UPV value of SSM was constructed by using the entire data of SSM incorporation of Na\( _2 \)SO\( _4 \) with SO\( _3 \) amounts ranging from 0 wt.% to 10 wt.% at the ages of 7, 14, 28, and 56 days, as illustrated in Fig. 8. The fitted curve of an exponential equation with empirical coefficients \( a \) and \( b \) of 0.08604 and 0.0154 was proposed, which was demonstrated by Equation 5. The equation had a coefficient of correlation (\( R^2 \)) of 0.975, which it could be concluded that a strong correlation was obtained [30].

\[
f_m = 0.08604e^{0.00154V_{UPV}}
\]  

(5)

4 Conclusions

Based on the experimental investigation that was conducted to investigate the mechanical strengths and UPV evaluation of supersulfated cement mortar containing sodium sulfate, the following conclusions may be drawn:

1. The incorporation of sodium sulfate, which acts as a sulfate activator in supersulfated cement, could enhance the 28-day compressive strength of hardened supersulfated cement mortar by about 2.94 to 4.26 times when compared to the mixture without sodium sulfate. The 28-day compressive strength of hardened supersulfated cement mortar with sodium sulfate with SO\( _3 \) amounts of 2.5 wt%, 7.5 wt%, and 10 wt% were approximately 50.2, 69.5, and 72.7%, respectively, of that OPC-based mortar.

2. The flexural strength of supersulfated cement mortar at 28 days increased about 2.89, 3.78, and 3.26 times for hardened supersulfated cement mortar with sodium sulfate with SO\( _3 \) amounts of 2.5 wt%, 7.5 wt%, and 10 wt%, respectively, when compared to the mixture without sodium sulfate.

3. The ultrasonic pulse velocity evaluation revealed that the hardened mortar mixture without sodium sulfate addition had very poor mortar quality. However, the addition sodium sulfate to the mixture as little as 2.5 wt.% could only improve to good mortar quality. Furthermore, higher sodium sulfate addition, such as 7.5 wt.% and 10 wt.%, could be improved to excellent mortar quality.

4. The correlation of hardened mortar compressive strength and the ultrasonic pulse velocity value has been successfully developed with strong correlation and a coefficient of determination (\( R^2 \)) of 0.975. The proposed equation could then be used for estimating the compressive strength of hardened supersulfated mortar from non-destructive test evaluation.

The present study proved that sodium sulfate could potentially be utilized as ground granulated blast furnace slag sulfate activator together with ordinary Portland cement as an alkali activator in producing eco-friendly supersulfated cement mortar. However, the hydration mechanism is still unclear. Therefore, further chemical and microstructural analysis could be performed as an extension of this study.

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