



Enhancing Mechanical Strength of Inorganic Geopolymer Using Phenol Resin

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ABSTRACT

The effect of organic phenol on the compressive and flexural strength of a metakaolin based geopolymer is studied. It was found that 12 wt% phenol resins can increase the compressive and flexural strength by 30% and 65%, respectively. In addition, the reinforcing effect of phenol resin is higher in Na-PS and Na-PSS type geopolymers rather than Na-PSDS. Two distinct endothermic peaks were detected in DTA curve which can be attributed to the release of water from phenol resin and its final thermal decomposition. The innovation of present research is the usage of water resistant phenolic resin as organic additive to geopolymer matrix.

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1. INTRODUCTION

The reaction of reactive aluminosilicate such as metakaolin and fly ash with highly concentrated alkali hydroxide or silicate solution produce amorphous aluminosilicate that was discovered first by Chelokovski in 1950. This material exhibits good properties such as high compressive strength, low shrinkage, designable setting time, acid resistance, fire resistance and low thermal conductivity. Therefore, these properties makes it a good candidate to replace traditional cementitious materials at low temperatures (up to 150°C). Another application for geopolymer materials is encapsulation of hazardous and toxic heavy metals (such as Cu, Cr and W ion) and nuclear waste [1-4].

Some researchers have used a variety of fibers [5-7] and particles [8, 9] to increase the compressive and flexural strength of the geopolymer matrix. Due to the 3D-cross-linked nature of formed aluminate, silicate and aluminosilicate species in setting and hardening reactions [10, 11] polymers are suitable for geopolymer matrix composites fabrication. However, little work has been done about organic-inorganic composite geopolymer materials. For example, Zhang et al. [12] used water-soluble organic polymers such as polyacrylic acid (PAA) sodium polyacrylate (PAA_{Na}), polyethylene glycol (PEG), polyvinyl alcohol (PVA), and

polyacrylamide (PAm) to investigate the effect of these materials on the compressive strength of a geopolymeric matrix. However, water susceptibility which restricts the extensive use of these materials is considered to be one of the negative properties especially in applications that water resistance is important. This is comparable with macro defect free (MDF) composites, which face the same problem when used with water-soluble organic polymers as a modifier [13]. Thus, for overcoming this problem in MDF fabrication, use of phenol resins has been introduced by Pushpalal et al. [14]. Their results show favorable effects of phenol resin on increasing the flexural strength of composites and also water resistance properties of materials.

This research focuses on investigating the effect of phenol resin on the mechanical strength of metakaolin based geopolymers, to examine the ability of phenol resins as a strength modifying agent for inorganic geopolymers. Thus, the main innovation of this paper would be usage of phenol resin as a water resistant binder instead of water soluble polymers which have negative effects on water resistivity of geopolymer base materials. This may improve the ability of geopolymer base materials as structural materials.

2. MATERIALS AND METHOD

WBB kaolin was used as a starting material and was calcinated in order to produce metakaolin. The Kaolin

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calcination was conducted at 750°C for 4 hours. Table 1 shows the chemical composition of kaolin used for the calcinations step. The specific surface area of metakaolin was 15 m²/g (BET method). The Phenol resin was of the resol type, produced under code IL-800 by Iran Resitan Company. Alcohol was used as a resin modifier. The resin/alcohol ratio was 0.40. An activating solution consisting of analytical grade sodium silicate solution, microsilica, and analytical grade of sodium hydroxide was used in order to obtain SiO₂/Al₂O₃, Na₂O/Al₂O₃, and H₂O/Na₂O molar ratios in geopolymer samples. Table 2 shows the oxide molar ratios of geopolymer specimens.

The desired activating solution was prepared of consisting of NaOH and Microsilica to the original sodium silicate solution. Then, phenol resin was mixed in to the active solution through use of a Hobart mixer. The last stage consisted of mechanical mixing of metakaolin with the active solution which contained the phenol resin. Initial curing was kept at 70°C for 3h and final curing occurred at 120°C for 10h in dry atmosphere. Measurements were done according to ASTM C39-36 for compressive strength and ASTM D790-86 for flexural strength. Thermal analysis experiments were done at a heating rate of 15°C/min.

3. RESULTS AND DISCUSSION

Experiments were set up to obtain optimum amount of phenol resin. First a geopolymer specimen with SiO₂/Al₂O₃=3.5 was adopted as the matrix, which according to some literatures [1-4] is one of the ratios that gives high mechanical strength. Then resin was added at the range between 6-18 wt% at even intervals. Figure 1 shows the effect of the added resin on the compressive strength of geopolymers.

It can be seen that the effective amount of phenol resin is 12 wt%. It seems that in smaller values, i.e. 6 to 10 wt%, the reaction between phenol resin and silicate and aluminate polymeric species in geopolymers did not occur completely or sufficiently. In higher amounts, i.e. 14 to 18 wt%, the amount of phenol resin was too high and the resultant paste was not stiff enough for molding and handling, i.e. its workability was low. It can be seen that even the pastes with 16 and 18 wt% phenol resin had no distinct consistency. Therefore, strength measurement testing was aborted. Consequently, 12 wt% phenol resin was adopted for the remainder of experiments.

Figures 2 and 3 show the effect of the addition of 12 wt% phenol resin on the compressive and flexural strength of geopolymer matrices with different SiO₂/Al₂O₃ molar ratios, respectively.

TABLE 1. Chemical composition of WBB kaolin. All values are in weight percent.

SiO ₂	Al ₂ O ₃	Na ₂ O+K ₂ O	Fe ₂ O ₃	TiO ₂	LOI
48.8	35.4	3	0.8	<0.1	11.6

TABLE 2. Oxide molar ratios of geopolymers

SiO ₂ /Al ₂ O ₃	Na ₂ O/Al ₂ O ₃	H ₂ O/Na ₂ O
2	1	11
3	1	11
3.3	1	11
3.5	1	11
4	1	11
4.5	1	11

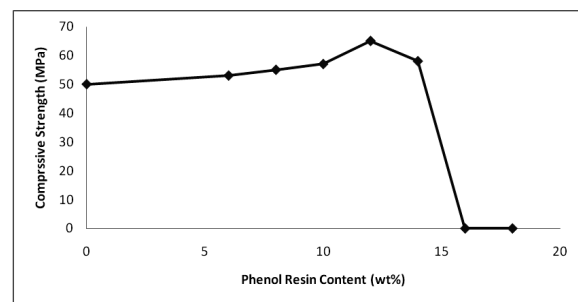


Figure 1. Optimum amount of phenol resin

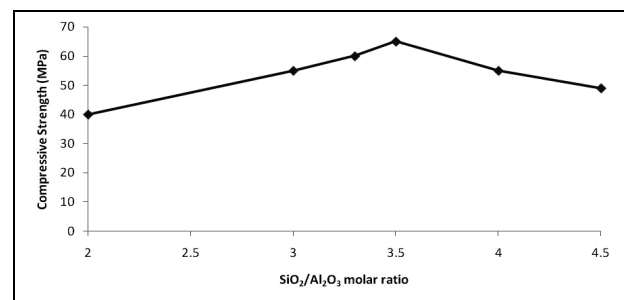


Figure 2. Effect of 12 wt% phenol resin on compressive strength of geopolymers

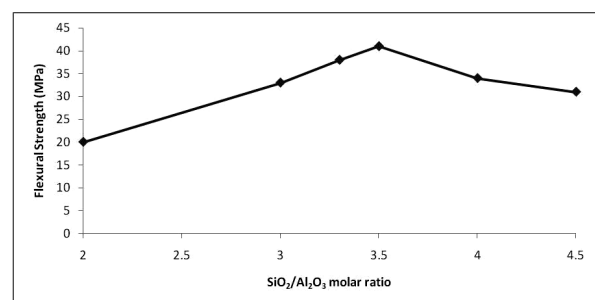


Figure 3. Effect of 12 wt% phenol resin on flexural strength of geopolymers

It can be seen that in both cases, the specimen with $\text{SiO}_2/\text{Al}_2\text{O}_3=3.5$ has maximum strength values. This is consistent with literatures [3], and [4] which shows that geopolymerization has occurred. Phenol resin enhances compressive and flexural strength of geopolymers by approximately 30% and 65%, respectively. It is interesting that the reinforcement effect of phenol resin was higher in Poly-Sialate (PS) and Poly-Sialate-Siloxo (PSS) than Poly-Sialate-Disiloxo (PSDS) geopolymers. Thus, reaction between PS and PSS geopolymers with the phenol resin occur faster and the reinforcement effect of phenol resin is higher in geopolymer matrices. Less stiffness and porosity of geopolymeric gel of PS and PSS type geopolymers can be attributed to this.

Figure 4 shows the XRD pattern of specimen with maximum mechanical strength.

The amorphous nature of composite system is obvious, which is expected due to amorphous nature of both geopolymer and resin.

Figure 5 shows the STA curves of geopolymer sample with $\text{SiO}_2/\text{Al}_2\text{O}_3=3.5$. The main endothermic peak around 170°C on DTA curve attributed to dehydroxylation reaction which is proportional to weight loss on TG curve.

According to Duxon et al. [4], structural densification of geopolymeric gel and viscous sintering can occur between 300 to 700°C .

Figure 6 shows the STA diagram of modified geopolymer with 12 wt% phenol resin specimen with $\text{SiO}_2/\text{Al}_2\text{O}_3=3.5$. TG curve shows two weight losses; the first attributed to water releasing from phenol resin that occurred about 100 - 150°C , and the second attributed to thermal decomposition of phenolic resin at 400°C . The exothermic peak revealed on DTA curve is proportion to those weights loss on TG curve.

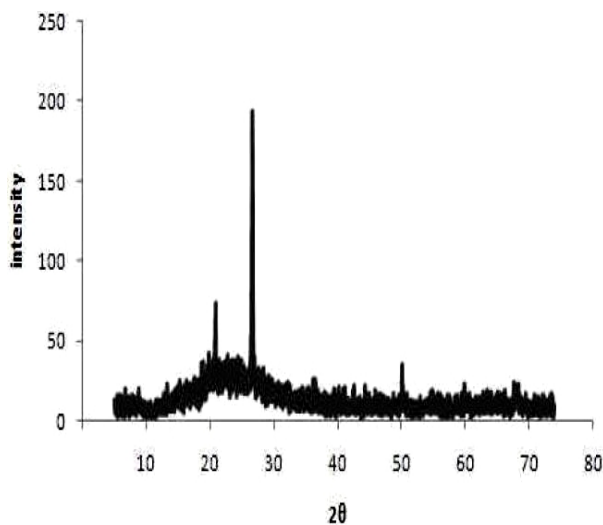


Figure 4. XRD pattern for specimen with $\text{SiO}_2/\text{Al}_2\text{O}_3=3.5$

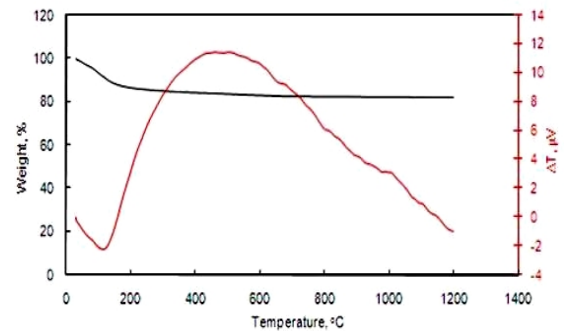


Figure 5. STA curves of geopolymer matrix with maximum mechanical strength

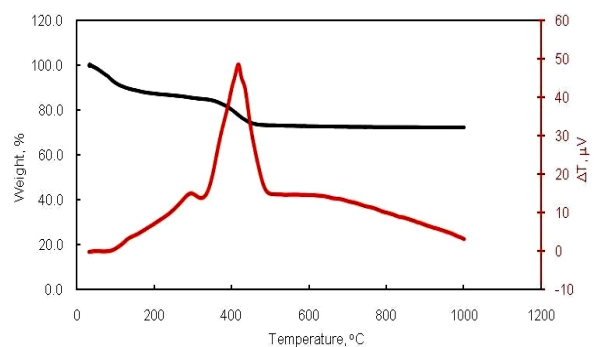


Figure 6. STA curves of geopolymer modified with 12 wt% phenol resin

4. CONCLUSION

Through the present research, it was shown that the compressive and flexural strength of geopolymers can be improved to 30% and 65%, respectively. The optimum amount of phenol resin for the present study was found to be 12 wt%.

Moreover, the reinforcing role of phenol resin is more effective in PS and PSS geopolymer due to less stiffness and porosity of the gel structure. The STA curve consisted of two weight losses and endothermic peaks which were attributed to the release of water and thermal decomposition of phenol resin.

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در این پژوهش ابتدا با افزودن رزین فنولیک به عنوان تقویت کننده به زمینه ژئوپلیمری، کامپوزیت ژئوپلیمر- رزین فنولیک ساخته شد. سپس به بررسی تاثیر رزین فنولیک بر استحکام فشاری و خمشی زمینه پرداخته شد. نتایج نشان داد که ۱۲wt% رزین فنولیک می تواند استحکام فشاری ژئوپلیمر را تا ۳۰٪ و استحکام خمشی آن را تا ۶۵٪ بهبود ببخشد. به علاوه، اثر تقویت کنندگی رزین فنولیک در ژئوپلیمرهای نوع پلی سیالات و پلی سیالات سیلوکسو بیشتر از نوع پلی سیالات دی سیلوکسو می باشد. آزمایش STA نشان داد که دو اتفاق حرارتی مهم در این کامپوزیت رخ می دهد که مشتمل بر آزاد شدن آب از رزین فنولیک و تجزیه حرارتی نهایی رزین می باشند. نوآوری پژوهش حاضر در استفاده از رزین فنولیک به عنوان افزودنی آلی مقاوم به رطوبت نسبت به پلیمرهای محلول در آب می باشد.

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