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THE EFFECT OF CHEMICAL FACTORS AND GLASS POWDER ON THE RHEOLOGICAL PROPERTIES OF METAKAOLIN BASED GEOPOLYMER PASTES

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Abstract: The production of geopolymer materials comprises an intricate chemical reaction between a solid aluminosilicate source and an alkali hydroxide or a silicate solution, which may result in an amorphous alkali-aluminosilicate product. Supplementary cementitious materials (SCMs) such as Metakaolin (MK) and Glass powder (GP) are materials rich of silica and alumina contents. However, the use of these materials in geopolymeric networks demands higher soluble content of silica and alkaline ions such as sodium (Na^+) and potassium (K^+). Variations in these soluble contents may strongly affect the rheological properties of fresh geopolymer paste. This study aimed to investigate the yield stress, plastic viscosity and shear stress of a geopolymer network composed of MK and GP as precursors, and sodium silicate and sodium hydroxide as alkaline reagents. The mix design technique used in this study was developed based on targeted ratios of SiO_2/Al_2O_3 and Na_2O/SiO_2. The rheological parameters of the fresh pastes were correlated with the chemical factors considered in the mix design procedure. The results show that the higher SiO_2/Al_2O_3 and Na_2O/SiO_2, the lower yield stress could be expected while plastic viscosity increased. Additionally, glass powder leads geopolymer mixture to the strong Bingham model fluidity and flow under lower water content, though more viscous and faster poly-condensed material.

1 INTRODUCTION

Concrete is the most popular and widely used construction material worldwide (Sabir, Wild, and Bai 2001). Current trends in the production of concrete increase the demand of primary raw materials, especially Portland cement (PC) which requires highly sophisticated energy in its production. By considering an estimated annual growth of 4% in cement production (Kusuma, Budidarmawan, and Susilowati 2015), the increase in CO_2 emissions causes additional environmental problems. Therefore, in the last decade, research trends moved toward modifying and/or reducing cement productions (Mikulčić et al. 2016; Cortada Mut et al. 2015; Madani Hosseini, Shao, and Whalen 2011; Abdul-Wahab et al. 2016; Vargas and Halog 2015). One of the interesting advancements made toward modifying the cement industry, the latest discovery on fire-resistance polymers by Joseph Davidovits (Davidovits 2015), which created a unique opportunity for cement and concrete researchers to focus on the use of geopolymers in 100% cement-free materials.

Geopolymers are chemically described as the reaction between a solid aluminosilicate source and highly alkaline solutions, leading amorphous 3D networks of alkali-aluminosilicate compound (Davidovits 2015; Duxson et al. 2006). The reaction is also known as geopolymerization, involving three different steps: 1- the dissolution of Si and Al from the precursors, 2- agglomeration of dissolved elements, and 3- poly-condensation of the dissolved components (De Silva, Sagoe-Crenstil, and Sirivivatnanon 2007).

Supplementary cementitious materials (SCMs) such as metakaolin (MK) and Type F fly ash are the most prominent resources of silica and alumina which can be taken account for geopolymerization. Davidovits (2015) categorized the geopolymeric networks into two different systems: conventional system with alkali reagents only, and K-Ca aluminosilicate system with the dissolution of sorosilicate compounds in the precursors. Therefore, SCMs and any material rich in alumina and silica, can be polymerized according to their nature and appropriate geopolymeric system.

In the past years, there have been intensive research background on geopolymer materials which have considerably improved the understanding of the geopolymerization process. The mechanical and microstructural properties of geopolymers have been investigated by considering chemical factors such as SiO_2/Al_2O_3, R_2O/SiO_2 and R_2O/Al_2O_3 ratios in which R is Na^+ or K^+ (Davidovits 2015; Singh, Gupta, and Bhattacharyya 2015; Dadsetan et al. 2019). For instance, Van Jaarsveld and Van Deventer (1999) found a clear relationship between the mechanical properties and R_2O/SiO_2 ratio. This has been later approved by other researchers using different precursor-based geopolymers (Xu and Van Deventer 2002; Alanazi et al. 2017). The effects of temperature and humidity on the geopolymerization have been also studied. The findings are almost similar in which temperature at around 60^oC-70^oC and relative humidity of 70% were shown to enhance the geopolymerization reactions leading to better overall properties (Najafi Kani and Allahverdi 2009; Rovnaník 2010; Heah et al. 2011; Yousefi Oderji, Chen, and Taseer Abbas Jaffar 2017). Although the current literature provided appropriate information about the chemical reactions and mechanical performance of geopolymers, there have been few studies carried out on the rheological behavior of SCM-based geopolymers. Indeed, rheology is the science of the flow resistance under shear force that enables the characterization of a fluid in many aspects such as workability loss, compaction and pumpability (Barnes, Hutton, and Walters 1989; Laskar and Bhattacharjee 2013). It is known that cement paste can perform one of the steady state flows: 1- Newtonian, 2- shear thinning and 3- shear thickening, depending on the colloidal, viscous and inertial interactions derived from electrostatic, hydrodynamic and between particle forces (Roussel et al. 2010). The few studies on the rheology of SCM-based geopolymers have shown that the geopolymer pastes are solid-liquid suspension-dispersion systems in which alkaline reagents are able to produce positive ions and attract negative ions from the precursors through electric charge phenomena (Romagnoli et al. 2014, 2012). Therefore, alkaline reagents and the solubility of the ingredients in precursors have significant effects on the rheological characteristics of fresh geopolymer pastes (Vance et al. 2014; Palacios and Puertas 2011). The interactions in geopolymerization process are highly relying on the solubility of the precursors in the alkaline environment. Another study on fly ash and slag-geopolymer concretes found a clear relationship between the initial setting time and SiO_2/Al_2O_3 ratio (Kashani, Ngo, and Mendis 2018). Favier et al. (2014) showed that metakaolin-based geopolymer paste has lower colloidal and inertial interactions due to the higher solubility of metakaolin ingredients in touch with alkaline reagents. However, this would reasonably fluctuate by using different types of precursors in geopolymerization process.

Glass powder (GP) as one of the aluminosilicate sources has recently attracted the attentions of researchers in geopolymer technology (Vafaei and Allahverdi 2017a; Balaguer Pascual, Tognonvi, and Tagnit-Hamou 2014; Zhang 2015; Torres-Carrasco et al. 2015). The satisfactory mechanical performance of glass powder in geopolymers have been already reported (Vafaei and Allahverdi 2017b). However, the rheological properties of this material are not yet studied in literature.

This paper investigates the relation between the different chemical factors involved in geopolymerization (SiO_2/Al_2O_3 and Na_2O/SiO_2 ratios) on the shear stress, plastic viscosity and yield stress of fresh metakaolin-based geopolymer pastes. Also, the effect of replacing 25% of total precursor weight by GP on the mechanical and rheological properties of MK-geopolymer system has been considered. Similar to MK-geopolymer compositions, the mix design procedure used to find the equivalent mixture proportion of MK-GP-geopolymer was based on targeted chemical ratios.

2 EXPERIMENTAL WORK

2.1 Materials

Metakaolin and glass powder were used as aluminosilicate resource precursors. The metakaolin was a high reactivity product supplied by Powerpozz Company, USA. The glass powder was a waste material obtained from Tricentris Company in Quebec, Canada. X-Ray fluorescence (XRF) analysis had been conducted on the precursors and the quantitative chemical compositions of both MK and GP are presented in Table 1. The specific gravity of powders were also measured according to ASTM C 188 (ASTM C188-17 2017) by using Le Chatelier flask. Sodium silicate and sodium hydroxide were considered as alkaline reagents in this study. Sodium silicate was a lab graded water glass with 27.5% SiO_2 and 8.5% Na_2O manufactured by Westlab company, Canada. Sodium Hydroxide beads (97% purity), supplied by VWR company, Canada, were used to prepare the NaOH solutions of 12 molarity.

Table 1: Chemical compositions (wt%) and physical properties of metakaolin and glass powder

Component	Metakaolin	Glass powder
SiO_2	55.74	73.08
Al_2O_3	38.07	2.43
Fe_2O_3	1.84	0.36
CaO	0.03	11.02
MgO	0.18	1.24
Na_2O	0.02	13.21
K_2O	0.27	0.68
TiO_2	1.47	0.07
LOI	1.17	0.00
Physical properties		
Specific gravity (g/cm^3)	2.4	2.45

2.2 Mixture design method and mixture proportions

After preparing NaOH solutions by distilled water and cooling down the liquid, both alkaline reagents were mixed and left 24 hours inside a laboratory fume hood to release any possible heat extracted from mixing sodium silicate and sodium hydroxide. The mix design method employed in this research was based on targeted molar ratios of SiO_2/Al_2O_3 and Na_2O/SiO_2, then the liquid to solid ratio was calculated accordingly. The weights of alkaline reagents have been selected by considering the targeted molar ratios and the precursors. As the proposed mixture design requires a selected NaOH solution, it was decided to fix it at 12 molarity. Figure 1 shows the flowchart steps of the mixture design method used. In total, 7 geopolymer mixtures were investigated including 6 MK-based geopolymer paste mixtures with two SiO_2/Al_2O_3 ratios (3.2 and 3.6) and three Na_2O/SiO_2 ratios (0.22, 0.26 and 0.3). To evaluate the effect of glass powder, an equivalent SiO_2/Al_2O_3 ratio of 4.4 was considered to ensure the polymeric chains of paste in hardened phase as 3.6 for metakaolin-based geopolymer. The glass powder was replaced by 25% of total precursor weight. To assess the equivalency of glass powder mixture, compressive strength measurements have been carried out at 7 and 28 days on 50 × 50 × 50 mm cubes. After casting, samples were placed in plastic bags and kept in similar curing conditions at room temperature. The Na_2O/SiO_2 ratio was kept equal to 0.22 for all geopolymer pastes. The mixture proportions and the chemical factors are given in Table 2. The mix codes shown in Table 2, are based on: a) name of precursor, b) SiO_2/Al_2O_3 and c) Na_2O/SiO_2 ratios. For example:

- MK-3.6-0.26: Metakaolin-based geopolymer paste with $SiO_2/Al_2O_3 = 3.6$ and $Na_2O/SiO_2 = 0.26$;
- MK-GP-4.4-0.22: 25% glass powder as metakaolin replacement in geopolymer paste with $SiO_2/Al_2O_3 = 4.4$ and $Na_2O/SiO_2 = 0.22$.

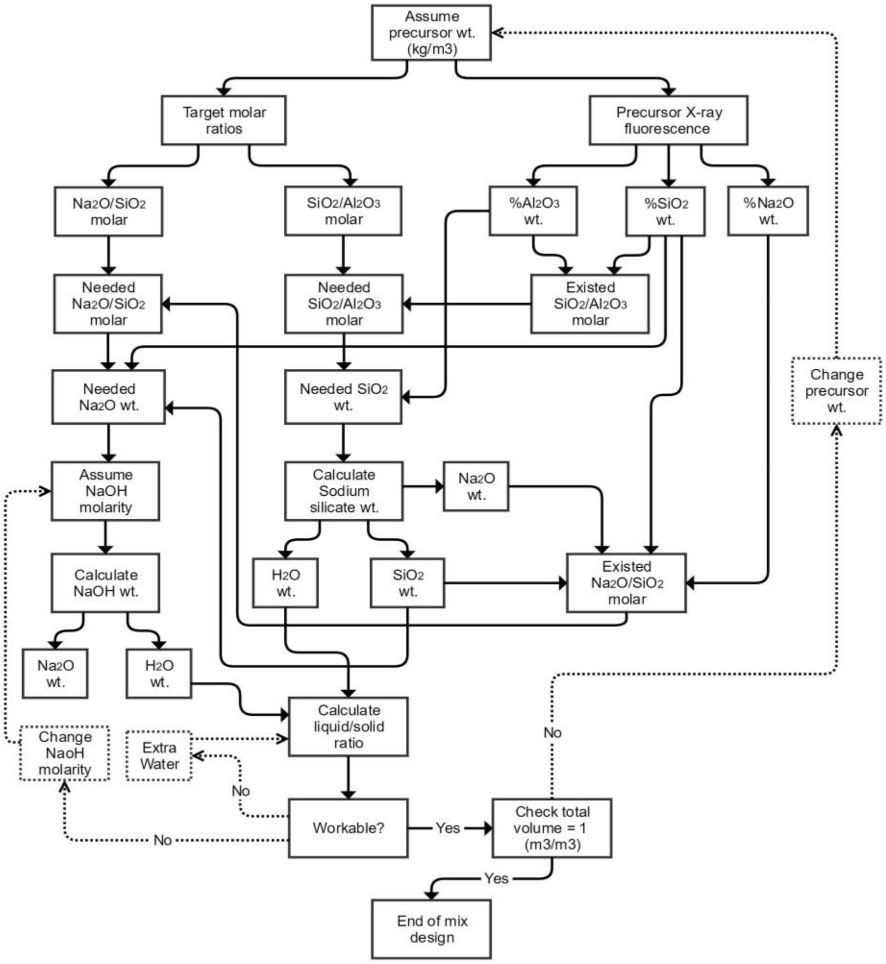

Figure 1: Flowchart of mix design method

Table 2: Mixture proportions (kg/m^3) and the chemical factors

Mix code	MK	GP	SS	NaOH	Extra Water	L/S*	SiO_2/ Al_2O_3	Na_2O/ SiO_2	H_2O/ Na_2O	Na_2O %
MK-3.2-0.22	770	-	450	311	90	0.55	3.2	0.22	21.00	6.18
MK-3.2-0.26	750	-	438	383	50	0.55	3.2	0.26	18.60	6.78
MK-3.2-0.3	730	-	426	450	10	0.55	3.2	0.3	16.71	7.32
MK-3.6-0.22	720	-	655	272	-	0.56	3.6	0.22	19.47	6.37
MK-3.6-0.26	670	-	610	333	-	0.60	3.6	0.26	18.53	6.95
MK-3.6-0.3	630	-	573	388	-	0.63	3.6	0.3	17.81	7.47
MK-GP-4.4-0.22	683	228	514	294	-	0.43	4.4	0.22	14.02	7.39

* L/S: Liquid to Solid ratio

2.3 Rheological characteristics

The rheological characteristics of 7 geopolymer pastes were evaluated by using a viscometer characterised with different shear rate levels and testing was performed at room temperature of about 22-25°C. The viscometer was able to record the torque T(Nm) at each rotational velocity N (rev/s). The flow resistance G (Nm) and relative viscosity H (Nm.s) were also measured by the device. As it was mentioned previously, the interactions in a suspension lead to a resistance under shear forces which can be simulated by Bigham model. The rheological parameters in this study were also measured by using Bingham fluid model as given in Equation 1. Plastic viscosity and yield stress were evaluated by using Reiner-Riwlin equations (Equations 2 and 3) (Feys, Verhoeven, and De Schutter 2008).

[1] $\tau = \tau_0 + \mu\dot{\gamma}$

[2] $\tau_0 = \frac{G}{4\pi h}\left(\frac{1}{R_i^2} - \frac{1}{R_o^2}\right)\frac{1}{\ln\left(\frac{R_o}{R_i}\right)}$

[3] $\mu = \frac{H}{(8\pi^2 h)\left(\frac{1}{R_i^2} - \frac{1}{R_o^2}\right)}$

where τ: shear stress (Pa), τ_0: yield stress (Pa), μ: plastic viscosity (Pa.s), $\dot{\gamma}$: shear rate (1/s), G: flow resistance (Nm), H: relative viscosity (Nms), R_i: the radius of inner cylinder (m), R_o: the radius of outer cylinder (m) and h: the height of cylinder (m). The rheological measurements were repeated at 7 different times after casting: 10, 30, 40, 50, 60, 70 and 80 minutes.

3 RESULTS AND DISCUSSION

3.1 Effect of chemical factors

The chemical factors involved in this study were SiO_2/Al_2O_3 and Na_2O/SiO_2 ratios targeted in the mixture design method described in Figure 1. To investigate the effect of each factor, 6 MK based geopolymer pastes were cast by using two SiO_2/Al_2O_3 (3.2 and 3.6) and three Na_2O/SiO_2 (0.22, 0.26 and 0.3). Plastic viscosity and yield stress of mixtures were measured at 7 different times after casting. The results are presented in Figures 2 and 3. From Figure 2-a, it can be seen that the lower value of Na_2O/SiO_2 (0.22) led to the highest yield stress. This is valid from the beginning up to one hour after casting, then the ratio of 0.3 increased dramatically at 70 minutes which caused a solid material. The increasing rate of yield stress in mixture containing Na_2O/SiO_2 = 0.26, represents a faster setting time than mixture MK-3.2-0.22. Plastic viscosity, on the other hand, increased at all times and by increasing Na_2O/SiO_2, as can be seen in Figure 2-b. Mixture MK-3.2-0.3 also achieved the highest plastic viscosity at all times up to 70 minutes, after casting. Additionally, Mixtures with SiO_2/Al_2O_3 of 3.6 (Figure 3-a) showed the same behavior in yield stress as for SiO_2/Al_2O_3 of 3.2, when increasing Na_2O/SiO_2 from 0.22 to 0.30. However, faster setting was observed in the mixture containing higher Na_2O/SiO_2 (0.3) after 50 minutes measurements. The variations in plastic viscosity shown in Figure 3-b, were also in the same order of plastic viscosity in Figure 2-b. This proves the higher Na_2O/SiO_2, the higher plastic viscosity can be expected at all times before setting of geopolymer pastes, and this despite of different SiO_2/Al_2O_3 ratios.

By comparing Figures 2-a and 3-a for different SiO_2/Al_2O_3 with the same Na_2O/SiO_2 ratios, all yield stress values decreased by increasing SiO_2/Al_2O_3 ratio from 3.2 to 3.6 at all times before setting. For instance, the yield stress for the mixture MK-3.2-0.26 at 10, 30 and 50 minutes in Figure 2-a, were 6.49, 9.33 and 14.56 Pa respectively. However, these values for mixture MK-3.6-0.26 in Figure 3-a, were 5.46, 8.56 and 10.62 Pa respectively. Therefore, it can be concluded that the higher SiO_2/Al_2O_3 ratio leads to the lower yield stress at all times before setting. Contrarily, the plastic viscosity increased by increasing SiO_2/Al_2O_3 ratio as can be seen in Figures 2-b and 3-b. This behaviour in metakaolin based geopolymers is in agreement with previous study carried out by Rovnaník et al. (2018). The low values of yield stress in this study can be explained by the negligible interactions between metakaolin particles and soluble alkaline silica in the geopolymer mixture (Favier et al. 2014).

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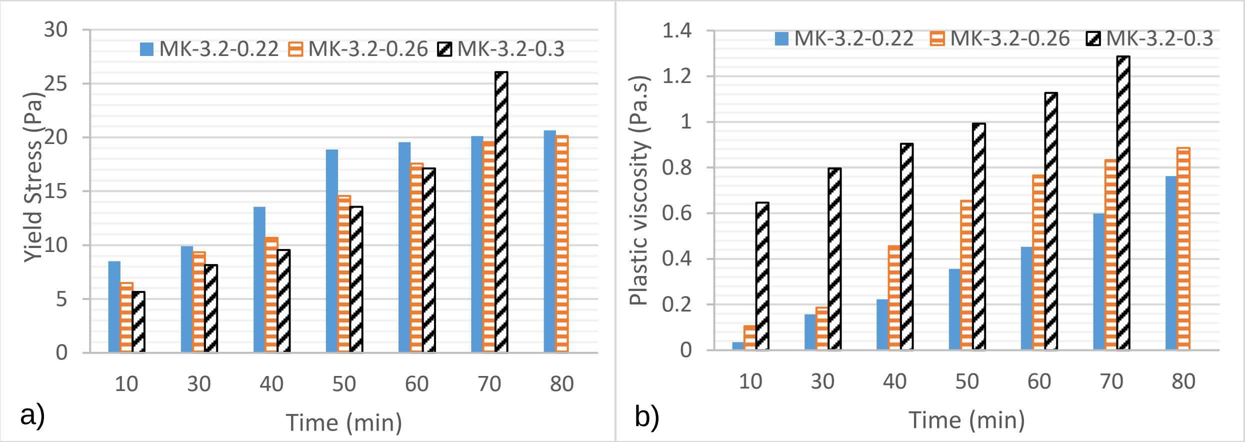

Figure 2 Rheological data for MK-3.2 with different Na_2O/SiO_2: a) yield stress and b) plastic viscosity

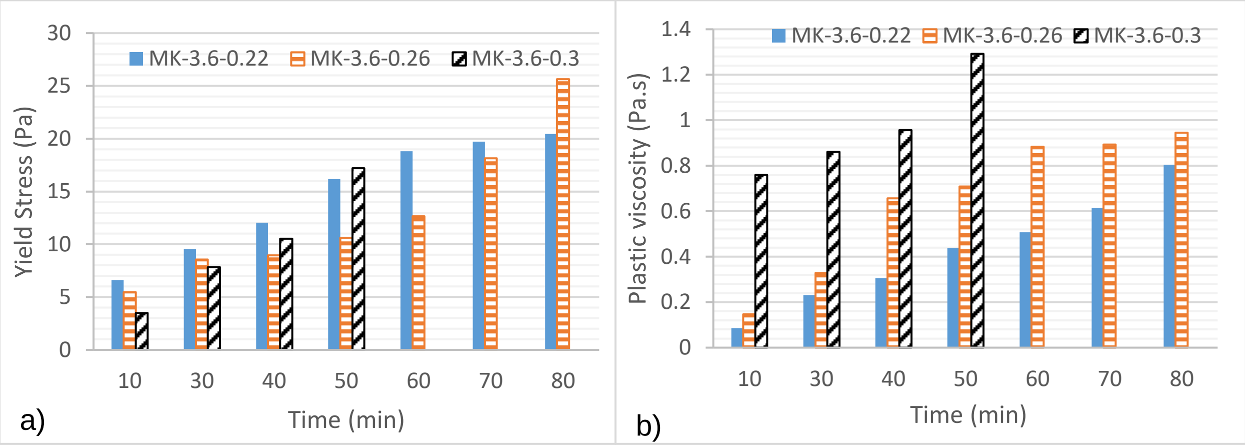

Figure 3 Rheological data for MK-3.6 with different Na_2O/SiO_2: a) yield stress and b) plastic viscosity

3.2 Effect of glass powder

As it was mentioned previously, an equivalent SiO_2/Al_2O_3 ratio of 4.4 was considered in order to evaluate the effect of glass powder in geopolymer mixture made by metakaolin. Glass powder was replaced by 25% of the weight of precursor in addition to SiO_2/Al_2O_3 of 4.4 and Na_2O/SiO_2 of 0.22 as the targeted ratios in mixture design procedure. The equivalent ratio refers to the equal polymeric chain of these materials in the geopolymerization process. The results are shown in Table 3. The values in Table 3 confirm that the addition of GP caused small increments in the compressive strength of MK-compositions, though the maximum difference between MK and MK-GP mixes was around 3.3 MPa at 7 and 28 days.

Table 3 Compressive strength results for MK and equivalent MK-GP based geopolymers

Mix code	Compressive Strength (MPa)	
	7 days	28 days
MK-3.6-0.22	49.16	52.34
MK-GP-4.4-0.22	52.40	55.63

Figures 4 and 5 display the flow behavior of geopolymer mixtures containing MK and MK-GP. The curves in Figure 4 indicate the slight Bingham model behavior with the low values of yield stress and plastic viscosity for MK-based geopolymer mixture. It can be noticed from Figure 4 that the flow resistance and consequent shear stress increased by time, however the shear stress values are very low even at 80 minutes after casting. This proves a higher setting time of the material, which can be explained by a longer time of geopolymeric reactions, especially coagulation and poly-condensation of the dissolved ingredients, known as low-viscous fluid.

The flow behavior of geopolymer containing 25% GP is shown in Figure 5. The flow curves present the strong Bingham fluidity of the mixture. The shear stress increased drastically by time, from 26.3 Pa to 175.2 Pa at maximum shear rates of 10 and 80 minutes after casting, respectively. This higher flow resistance of MK-GP mixture can be explained by the lower liquid/solid ratio of 0.43 compared to that of MK mixture with 0.56. The values of liquid/solid ratio are varied due to the initial workability of the mixtures. MK-3.6-0.22 was able to flow at the minimum L/S ratio of 0.55. NaOH molarity was selected as 12 molarity to avoid any value higher than 0.56 which lead to higher water content and probable interference in geopolymerization process. On the other hand, glass powder demanded lower L/S ratio for being workable. This is probably because of the lower specific surface area of GP particles compare to that of MK. Therefore, the lower L/S ratio increased the flow resistance of the mixture with the equivalent SiO_2/Al_2O_3 and the constant Na_2O/SiO_2 ratios.

Additionally, the changes in yield stress and plastic viscosity of both MK and MK-GP mixtures are shown in Figure 6. Yield stress values in Figure 6-a, for MK mixture increased from 6.6 Pa to 20.4 Pa at 10 and 80 minutes after casting, respectively. On the other hand, MK-GP mixture performed differently by a considerable increment from 15 Pa to 127.2 Pa. The analysis of the plastic viscosity variations in Figure 6-b, also shows the same trend of dramatic increase in MK-GP mixture (3 Pa.s to 13 Pa.s) than MK mixtures (from 0.09 Pa.s to 0.8 Pa.s). Taking into account the yield stress and plastic viscosity values of the tested mixtures, it can be stated that glass powder enables geopolymer mixture to flow under lower L/S ratio, though more viscous and faster poly-condensed material.

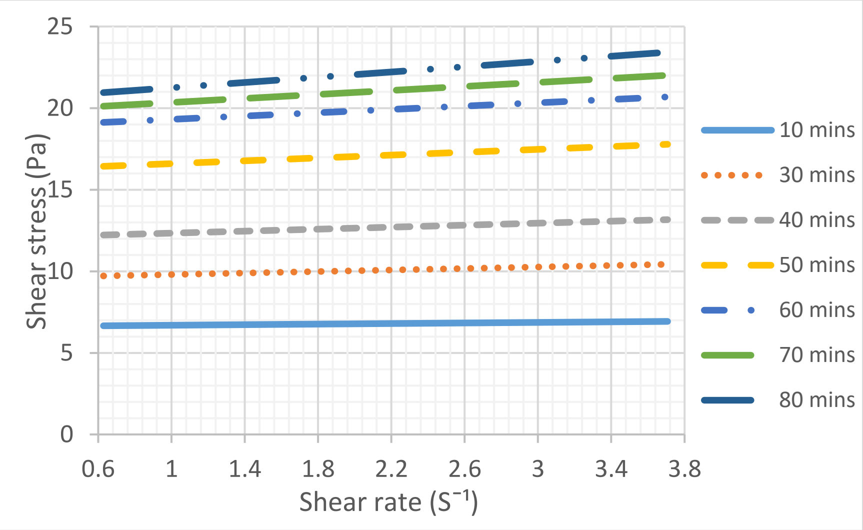

Figure 4 Flow curves of metakaolin-based geopolymer: MK-3.6-0.22

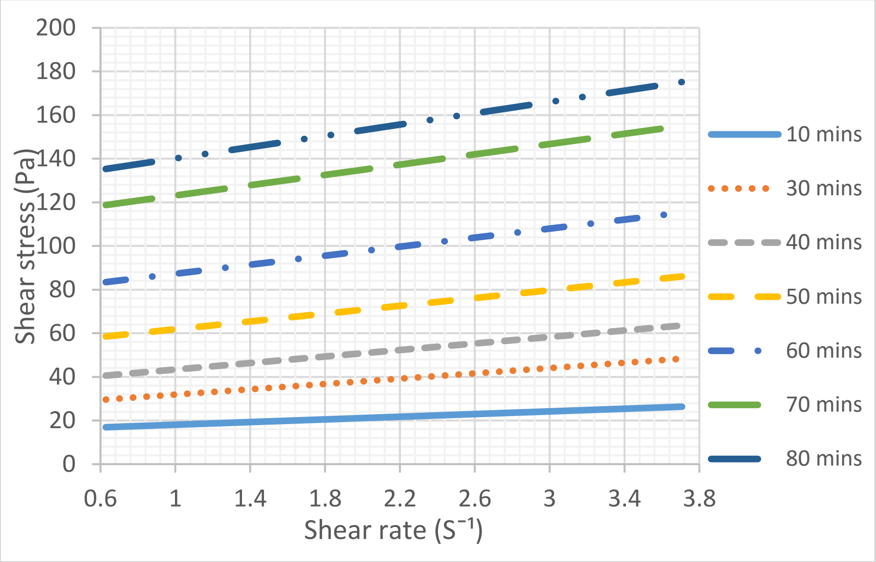

Figure 5 Flow curves of equivalent glass powder and metakaolin geopolymer: MK-GP-4.4-0.22

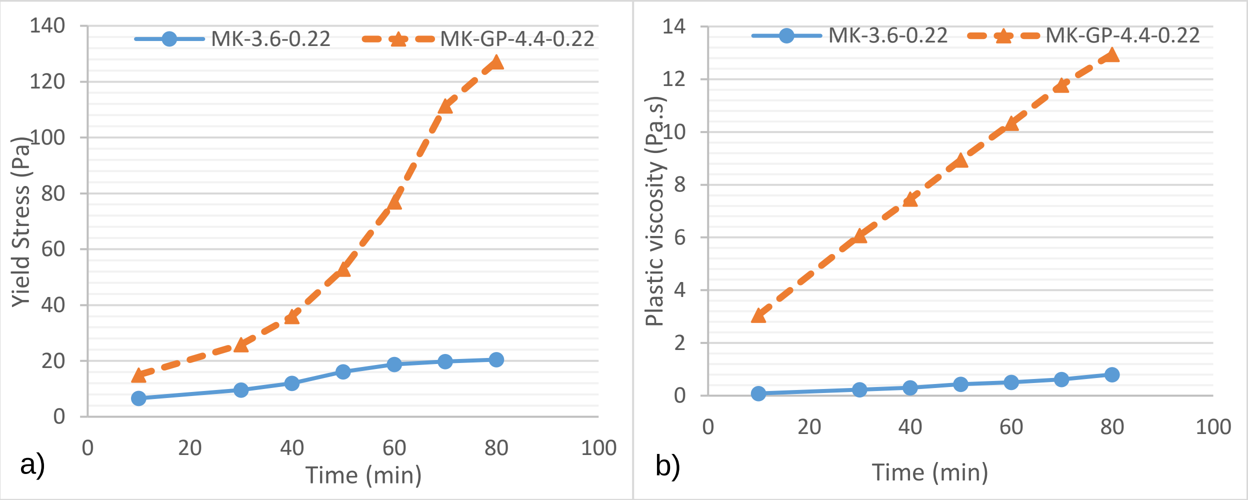

Figure 6 Changes in the rheological data of geopolymers over time: a) yield stress and b) plastic viscosity

4 CONCLUSIONS

According to the results obtained from this study, the primary conclusions can be summarized as follows:

1. Through the investigation on the effect of Na_2O/SiO_2 ratio on the metakaolin-based geopolymer, yield stress decreased by the increasing in the ratio, however the increasing rate of yield stress over time was more considerable in higher Na_2O/SiO_2 ratio. On the other hand, the higher Na_2O/SiO_2, the higher plastic viscosity can be expected at all times before setting for geopolymer pastes with the same SiO_2/Al_2O_3 ratios.

2. By increasing SiO_2/Al_2O_3 ratio, yield stress decreased at all times before setting while the plastic viscosity increased. This can be explained by the negligible interactions between metakaolin particles (alumina source) and soluble alkaline silica in the geopolymer mixture.

3. The low values of shear stress for metakaolin-based geopolymer compared to the equivalent glass powder mixture, proves the higher setting time of the material which can be explained by the longer time of the geopolymeric reactions, specifically agglomeration and poly-condensation of the dissolved ingredients,

known as low-viscous fluid. Contradictorily, the replacement of glass powder in metakaolin based geopolymer, leads to the strongest Bingham model fluidity of the mixture.

4. The analysis of the yield stress and plastic viscosity values of the tested mixtures confirmed that glass powder enables geopolymer mixture to flow under lower L/S ratio (lower water content), though more viscous and faster poly-condensed material are expected.

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