Properties of high density silica fume-based gel and its potential use in high temperature lubricants and geopolymer binders

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Abstract

In the present study, undensified silica fume reacted with a 10M sodium hydroxide solution to produce the sodium silicate gel having a pH of 14. The calorimetry of the reaction was evaluated. The gel was dried to increase its density by almost 35%. Thereafter, the thermogravimetry of the gel was performed at temperatures up to 1000°C. Except the loss in mass caused by the departure of physical and chemical water at temperatures below 200°C, no other mass loss process was identified in the gel. The potential applications of the gel as high temperature lubricants for automotive and metallurgical engineering and as alkaline activator in geopolymer binder was discussed. Since silica fume is a by-product material that do not require heating or grinding, and can react with sodium hydroxide solution at ambient temperature, the results suggested potential for enhanced sustainability in the reduction of embodied energy for the production of the materials like high temperature inorganic lubricant and alkaline activator for geopolymer binder.

Key words: Silica fume· Sodium silicate· Thermal properties· High temperature lubricants· Geopolymer· Sustainability

Introduction

Following the Kyoto Protocol aimed at tackling the problem of global climate change, the year 2020 was set as a target for several countries to lower considerably their anthropogenic Green House Gas (GHS) emissions. Carbon dioxide (CO_2) is the major GHG. The better use of existing technologies and the promotion of those that reduce CO_2 emissions is critical [1]. Soluble silicate glasses, liquids and solids are among the largest volume synthetic chemicals, surpassed in volume only by commodity acids and bases [2]. Sodium silicate ($Na_2O(SiO_2)_x$),

where x is the molar ratio, defining the number moles of silica (SiO_2) per mole of sodium oxide (Na_2O), also called water glass is one of those silicates. The product has a wide variety of uses, including the formulation of special cements for concrete, passive fire protection, textile and lumber processing, manufacture of refractory ceramics, adhesives and inorganic lubricants [2, 3]. In industry, the manufacture of sodium silicate is based on 4 steps: (i) the firing of a mixture of sodium carbonate (Na_2CO_3) and natural quartz sand (SiO_2) in an appropriate furnace at temperatures between 1400 and 1500°C according to equation 1, where a "solid glass" is obtained, (ii) the dissolution in water of the "solid glass" in a reactive vessel under high pressure and temperature, (iii) the filtration of the solution depending on the desired purity and (iv) the evaporation of the water to obtain the desired density of the solution [4]. This process is considered costly and energy intensive. It is also leads to air pollution by the emission of greenhouse gases such as carbon dioxide (CO_2) [4]. As an alternative to this process, other production methods were developed, based on the reaction of amorphous silica from various sources (rice husk ash, corncob, sugar cane bagasse ash) with aqueous sodium hydroxide [5-7]. But, less attention has been paid to silica fume which is known as one of the best source of amorphous silica with excellent results in concrete technology as a supplementary cementitious material (SCM) [8, 9].
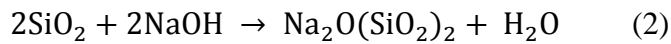
Silica fume (SF) is a by-product of the silicon and ferrosilicon industry. The reduction of high purity quartz to silicon at temperatures up to 2000°C produces SiO vapour, which oxidizes and condenses in the low temperature zone to ultra-fine particles consisting of 85 to 98.5% non-crystalline silica, also known as un-densified silica fume [10, 11]. The surface area of the particles varies from 13,000 to 30,000 $kg\ m^{-2}$. Silica fume is a high reactive pozzolanic material having particles that are 100 times smaller than the average cement particles [11]. When the un-densified silica fume is collected, it is very light with a bulk density in the range of 120 to 220 $kg\ m^{-3}$. For stocking space, the reduction of transport cost and dust pollution reasons, the un-densified silica fume is treated to increase its density within the range of 400 to 720 $kg\ m^{-3}$ [12]. This treatment of bulk density is usually accomplished by tumbling silica fume particles in a silo, which causes surface charges to build up. These charges draw the particles together to form agglomerates, which not only increase the density but also increase of embodied energy of the final material [13].

The main objective of the present study is to produce and characterize a high-density sodium silicate gel having a high pH with potential applications as high temperature lubricants for automotive and metallurgical engineering and as alkaline activator in geopolymer binders. Un-densified silica fume was used to produce a sodium silicate gel based on equation 2. Chemical

and mineralogical analyses, pH and density measurements, isothermal calorimetry and TG-DTG analyses were the tests performed on the raw material and products. The potential applications of the gel as high temperature lubricants for automotive and metallurgical engineering and as alkaline activator in geopolymer binder was discussed

$$xSiO_2 \ + \ Na_2CO_3 \ \rightarrow Na_2O(SiO_2)_x \ + \ CO_2 \qquad (1)$$
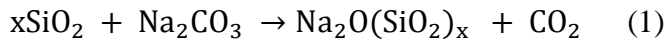
where "x" can be a fractional number.

$$2SiO_2 + 2NaOH \ \rightarrow \ Na_2O(SiO_2)_2 + \ H_2O \qquad (2)$$

Materials and methods

Materials and their characteristics

The raw materials used in the study consisted of 97.1% purity un-densified silica fume (USF) supplied by Elkem Silicon Materials in Norway and a 10M sodium hydroxide (NaOH) solution obtained by dissolving 400 grams of laboratory grade 97 % purity NaOH pellets in de-ionized water. The NaOH solution was prepared 24 hours before its use and kept in a container with a lid. The chemical composition of the silica fume sample was determined by X-Ray Fluorescence (XRF) spectrometry, using a Bruker S4 Pioneer wavelengths dispersive (WD-XRF) spectrometer. The mineralogy by X-ray diffraction (XRD) analysis of that sample was carried out under room temperature using a STOE company Powder Diffraction System with operating conditions of CuKα sealed tube and radiation wavelength of 1.54060 nm.

Characteristics of USF in Table 1 revealed that, SiO_2 was the major constituent oxide; with minor other oxides such as Al_2O_3, MgO and Fe_2O_3. The XRD pattern in Fig. 1 showed that USF was essentially in an amorphous state with some quartz impurities.

Table 1 Chemical composition and specific gravity of USF

Oxide (%)	USF
CaO	–
SiO_2	97.1
Al_2O_3	0.1
MgO	0.15
Fe_2O_3	0.2
MnO	-
S^{2-}	-
SO_3	0.06
K_2O	-
N_2O	-
L.O.I.	-
Specific gravity (g cm^{-3})	2.20
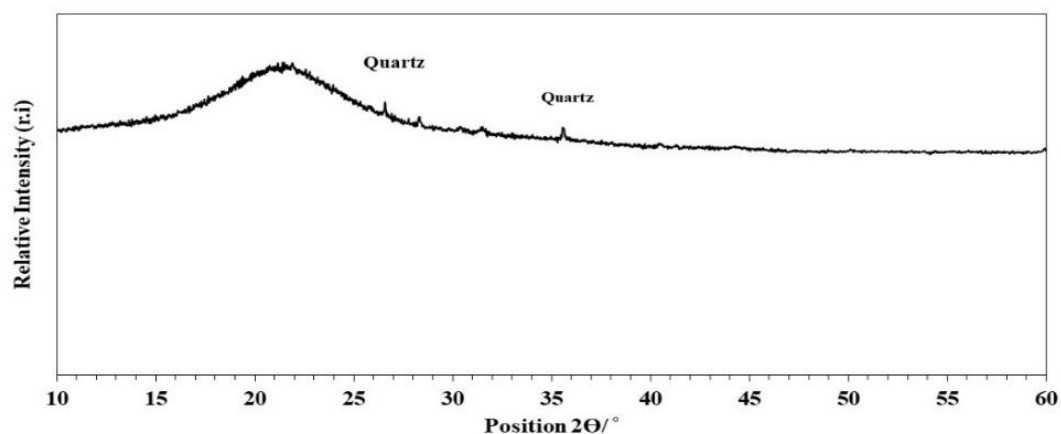
Fig. 1 XRD pattern of USF

Methods

Preparation of the USF gel

The chemical equation 2 was used as the basis for the calculation to obtain the sodium silicate gel with SiO_2/Na_2O ratio = 2. According to equation 2 and considering the purity of USF which was 97.1 % SiO_2, an equivalent of 123.58 grams of USF was mixed per 200 mL of the 10M

NaOH solution. The proportion of the constituents in the mixture was multiplied by 10 to obtain the desired quantity of the gel, which was thereafter left to react for 48 hours in a closed container at a room temperature of $20 \pm 1°C$ before use. After the reaction period, the density of the gel was measured by determining the mass of a given volume (40 to 60 mL) of the gel. Its pH was measured using a digital pHmeter. The gel was thereafter left to dry for 24 hours in an oven at 105°C using a flat container. Its density was measured once more. Fig. 2 shows the aspect of the dried gel.

Fig. 2 The dried USF gel

Characterization techniques

The calorimetry of the reaction between USF and the 10M sodium hydroxide solution was investigated for 24 hours using the isothermal calorimetry by a Tonitechnik device. Thereafter, test specimens of USF and its based gel were submitted to thermogravimetric analysis (TG-DTG) at up to 1000°C to study their behaviour at elevated temperatures using a TA Instruments TGA 55 equipment operating with the argon gas at $10°C$ min^{-1} heating rate. The mass of the testing sample for TG ranged from 4 to 10 mg.

Results and discussions

PH and densities of the gel

The pH and the density values of the gel are presented in Table 2.

Table 2 PH and density values of the USF based gel

| Designation	pH	Density before drying (g mL^{-1})	Density after drying (g mL^{-1})
USF based gel	14	1.52±0.01	2.05±0.02

The pH values of the gels are higher than that of commercial sodium silicate solutions, which ranges between 10 and 13. This could be due to un-reacted sodium hydroxide solution present in the medium. The higher pH of the gel could contribute positively to its stability, since lowering the pH leads to condensation reactions, thus a change in the silica speciation [14]. The increase in the density of the gel during the drying process is a result of the evaporation of part of the water in the gel, which also increased its viscosity. The increase in the density of the gel by almost 35% could contribute to ease the transportation of the material.

Isothermal calorimetry of the reaction between silica fume and sodium hydroxide

The isothermal calorimetry was used to evaluate the kinetic of the dissolution of silica fume in the 10M sodium hydroxide solution during 24 hours. According to equation 2, 5.5 mL of the 10M sodium hydroxide solution reacted with 3.40 g of USF. Fig. 3 and Fig. 4 present the recorded data for 24 hours of reaction.
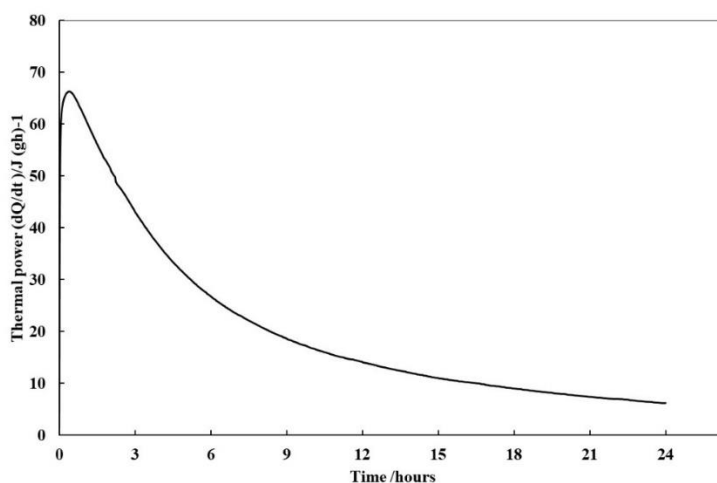
Fig. 3 Thermal power generated during the exothermic reaction of USF and sodium hydroxide

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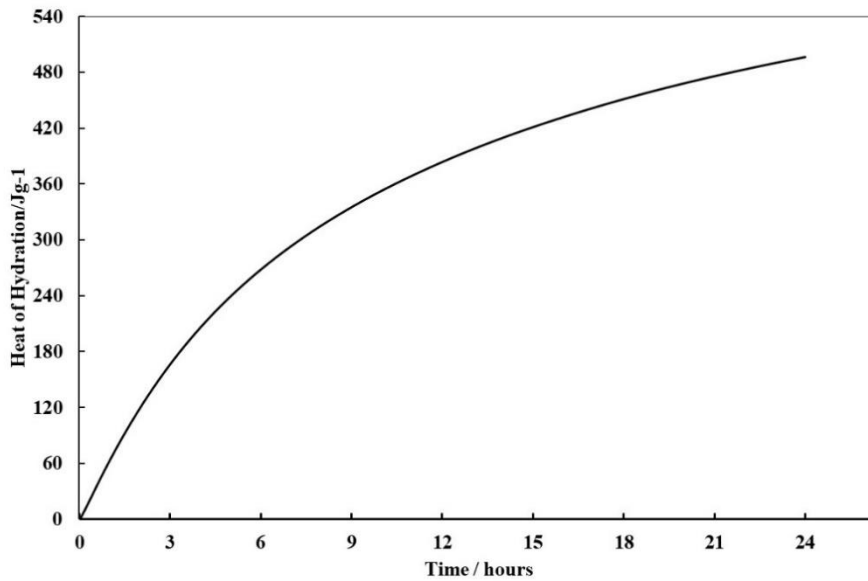
Fig. 4 Cumulative heat flow during the reaction of USF and sodium hydroxide

It was shown that the dissolution reaction, which is exothermic, started just after silica fume and sodium hydroxide were in contact. The maximum thermal power was achieved at about 20 minutes after the beginning of the reaction. The maximum heat flow was not reached after 24 hours of reaction, suggesting that although most of silica species are rapidly dissolved at the beginning of the reaction, a residual dissolution of silica species continued. In fact, during the reaction of silica in the presence of a basic solution, depending on its concentration, there is formation of SiO^- ions in the silica framework, which facilitate the detachment of Si in the network. The dissolution rate of silica is extremely high when silica is in an amorphous state and pH higher than 9 [15, 16, 17]. The high pH of the medium contributed the formation of silicate species and to the rapid dissolution of silica.

Thermogravimetric analyses of USF and its based gel

The thermogravimetric analysis measures the mass change of sample due to the increase in temperature, because of the decomposition reactions producing gaseous compounds. USF was subjected to the analysis as indicated in Fig. 5.

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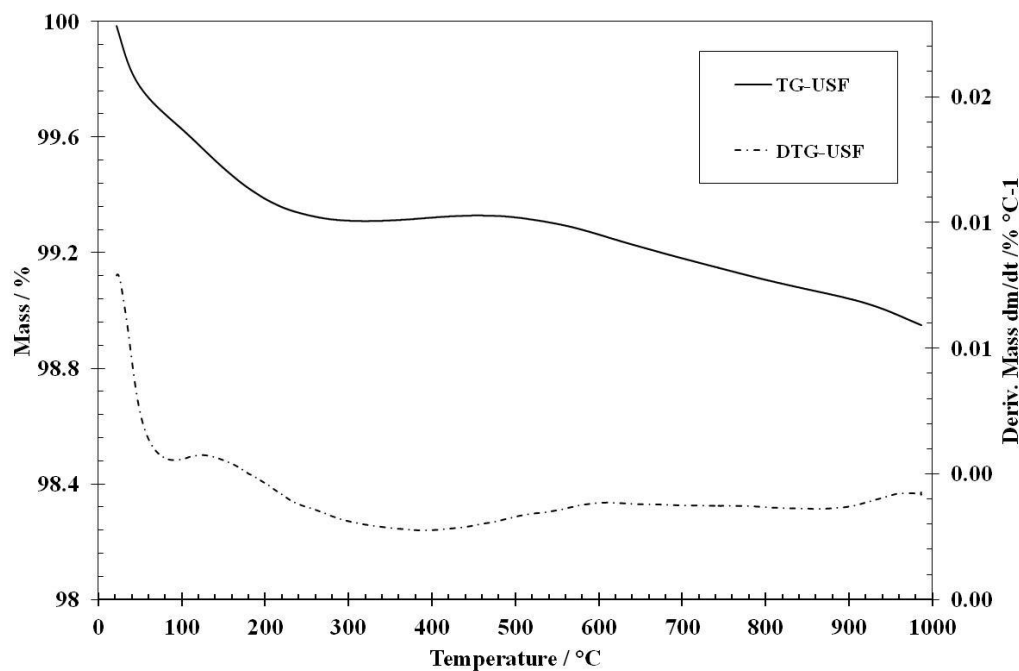
Fig. 5 TG-DTG curves of USF

As the heat increased from ambient temperature to 1000°C, USF exhibited slight mass loss of 0.96 % essentially due to loss of the remaining water in the sample. A stable trend during the heating at temperatures higher than 100°C was presented by the sample, showing that, except for the loss of evaporable water, no other decomposition and hence no further associated mass losses occurred in the sample.

When the USF gel was subjected to TG-DTG measurements, the result in Fig. 6 showed that, except for the loss of evaporable, physical and chemical absorbed water which happens at temperature below 200°C, no mass loss was detected in the sample at up to 1000°C. Subasri and Naife [18], who studied the phase evolution on heat treatment of sodium water glass with SiO_2/Na_2O ratio of three, stated that, phase change might happen in the sample. They reported that the water glass, initially in an amorphous solid state, starts to form crystals of β-$Na_2Si_2O_5$ as layered sodium silicate at about 400°C and crystallizes the SiO_2 modification cristobalite at 600°C that co-exist along with β-$Na_2Si_2O_5$ up to 700°C. At 750°C, $Na_6Si_8O_{19}$ appears as a separate phase and beyond that temperature, the system turns into liquid.

Layered sodium silicates among which β-$Na_2Si_2O_5$ were reported to be useful as high temperature inorganic lubricants [19]. Consequently, the elaborated gel could serve as raw material for high temperature inorganic lubricants used in automotive and metallurgical engineering.

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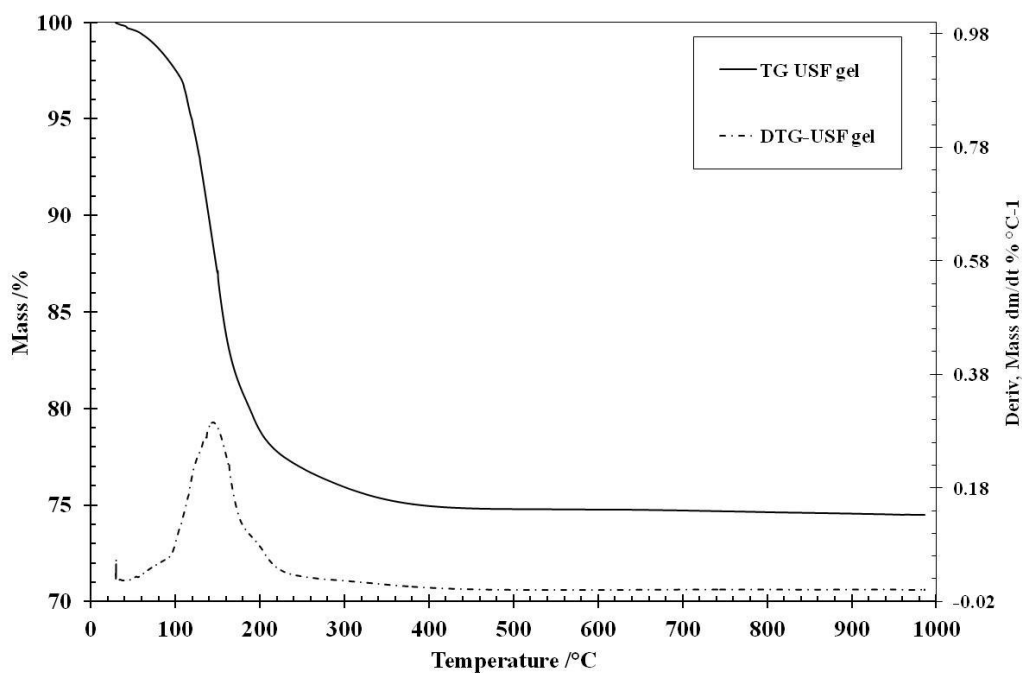
Fig. 6 TG-DTG curves of USF based gel

Organic materials such as fats, oils and soaps are generally used as lubricants. However, in the aerospace and power generation sectors and the automotive engineering and metal processing industries, key frictional components in machineries are required to operate at temperatures above 300°C, where organic lubricants cannot perform. It was reported that friction can cause wear and energy dissipation directly or indirectly responsible for about one-third of the world energy resource consumption [19]. Several inorganic polymers have shown good performances in high temperature friction operations [20]. Among them, silicate glasses which have been suggested in several tribology applications. Layered sodium silicates were reported to provide effective wear protection because of their layers characteristics, similar to standard inorganic lubricants like MoS_2 and graphite [19, 21, 22]. Ordinary sodium silicate is water soluble and less stable at high temperatures compared to layered sodium silicates, which are water insoluble [19], and more stable at high temperature after wear. They are a group of materials with complex polymorphism. Depending on the temperature, pressure and synthesis composition, the most well-known layered sodium silicates are α-$Na_2Si_2O_5$, δ-$Na_2Si_2O_5$, β-$Na_2Si_2O_5$ and kemanite ($NaHSi_2O_5.3H_2O$). They can be used as solid lubricants or dispersed in oils like oleic acids and liquid paraffin. When α-$Na_2Si_2O_5$ was used as additive in liquid paraffin and 150SN base oil (mineral base oil), it considerably improved maximum non-seizure load (P_B) from 392N to 470N and from 392N to 647N respectively [19]. In practice, standard α-$Na_2Si_2O_5$, δ-

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$Na_2Si_2O_5$, and β-$Na_2Si_2O_5$ are obtained by roasting ordinary sodium silicate at temperatures between 600°C and 780°C [19]. Further investigations could consider the study of the tribological properties at high temperatures, of the fired product of the elaborated silica fume based gel. From the thermogravimetry information, further synthesis of layered sodium silicates could be obtained by thermal treatment of the elaborated gel within the temperature range of 200°C to 800°C, with the aim of lowering the synthesis temperature and studying the wear and friction characteristics of the elaborated materials.

Sodium silicate was also reported as a major constituent of alkaline activators for geopolymer binders [23]. The geopolymer binder, which derives from the polycondensation of amorphous and three-dimensional aluminosilicate precursors in the presence of alkaline activator solutions [24, 25], was presented in numerous studies as a prominent Portland cement substitute in some applications with additional benefits in thermal insulation and fire resistance [26-28]. The concern was about the high level of carbon dioxide (CO_2) emission during Portland cement production, which was estimated at about 5 to 7% of the global anthropogenic CO_2 emission. Although some studies stated that geopolymer binders could lower the amount of greenhouse gases emission by 20 to 80 % compared to Portland cement [29-33], others indicated that the CO_2 emission could be reduced by 9% [34]. In all the cases, lower reduction figures where obtained when waste and by-products were used in the production process involving less energy demand. The alkaline activator solution commonly used in the geopolymer binder production was a mixture of sodium silicate and sodium hydroxide [35]. The major component responsible to CO_2 emission in the geopolymer production was sodium silicate production and its treatment, which accounted for 50% in volume of the environmental impact [5]. The elaborated sodium silicate gel was produced from silica fume and densified at 105°C. It has the potential to contribute significantly to the reduction the embodied energy and greenhouse gas emission of the resulted geopolymer binder made with the activator solution utilizing the elaborated gel. Consequently, the sustainability of geopolymer binders could be improve. While industrial waste ashes are re-utilized to produce geopolymer at ambient temperatures, the amount of greenhouse gas emitted to the environment might be lowered by 26 to 80 % compared to Portland cement [36-40]. Further study could determine the dissolution mechanism of the densified silica fume-based sodium silicate gel in water and the properties of geopolymer paste and concrete made with this solution.

Conclusions

This study focussed on the determination of thermal, physical and chemical characteristics of a high-density sodium silicate gel obtained by the reaction of silica fume and 10M sodium hydroxide solution. Potential applications of the elaborated gel were provided. From the findings, it was concluded that:

1. The density of the gel increased by almost 35% after drying at 105°C for 24 hours. This could significantly contribute to reduce the volume of the material during transportation;

2. The dissolution of silica fume in 10M sodium hydroxide solution was a rapid process attaining which its maximum dissolution rate at about 20 minutes after the reactants were in contact, yielding a solution with pH of 14;

3. Except for the mass loss due to the evaporation of different types of water in the gel, no other mass loss were observed in the material at up to 1000°C;

4. The gel possesses potential for use as raw material for layered sodium silicate for high temperature tribological application in automotive and metallurgical engineering, and as alkaline activator for geopolymer binders;

5. Since the silica fume is a by-product material that did not require to be heated or ground, and could be reacted with the sodium hydroxide solution at ambient temperature, the results suggested potential for enhanced sustainability in the reduction of embodied energy when applied in automotive and metallurgical engineering, or as alkaline activator for geopolymer binders.

Further research will concern the determination of the wear and friction properties at temperature higher than 300°C of the product of thermal treatment of the gel between 20 and 800°C. Investigation will also done on the degree of conversion of reactants, the amount of free water, the viscosity of the gel, its dissolution mechanism in water and properties of geopolymer binders made with the gel as based alkaline activator.

Acknowledgements

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Authors contribution

Ndigui Billong: Conceptualization, investigation, methodology, writing of original draft;

Jonathan Oti: Conceptualization, project administration, funding acquisition, writing, review and editing;

John Kinuthia: Conceptualization, Supervision, review and editing;

Rukshane Navaratne: Conceptualization, review and editing;

Jiping Bai: Conceptualization, review and editing.

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There is no conflict of interest about this research