



Temperature-Dependent Classification of Geopolymers Derived From Granite Designed for Well Cementing Applications

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Alternative materials such as geopolymers appear to have potential advantages compared to Portland cement. However, the application of geopolymers for all sections of the well is still a major challenge due to the difference in temperature ranges. To that end, the classification of the granite-based geopolymer mix designs requires a thorough investigation of various properties at a range of different operational temperatures. In this study, three mix designs are presented for different well sections at temperatures ranging from 5 °C to 60 °C. The mix designs for low temperatures (<50 °C) were tuned by adding CaO to the dry solid blend. Workability, rheology, short-term compressive strength, and X-ray diffraction (XRD) analysis were conducted to conclude the performance of the mix designs under study. Results highlight the presence of Ca content (wt%) in mix designs and its role in enhancing material performance at low operational temperatures. The study reveals a promising future application of the granite-based geopolymer for well construction and abandonment at varying depths with recommendations for further improving the performance by the addition of chemical admixtures. In addition, the relation between temperature and Ca content was highlighted, and more investigations into the kinetics governing these two parameters were recommended. [DOI: 10.1115/1.4063027]

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Introduction

Ordinary Portland cement (OPC) has been widely used as a zonal isolation material in the petroleum industry for establishing annular barriers and cement plugs. As a hydraulic binding material, OPC has undergone extensive research on its chemical admixtures and modifications to its chemistry, making it a practical choice for well cementing operations. The industry is well aware of the existing limitations of Portland-based cement, including concerns related to its long-term durability, low flexibility, and leaching when exposed to downhole chemicals, which can result in well integrity issues [1–3]. Moreover, with the global movement toward sustainability, OPC is not quite ideal to be continuously used due to its high environmental cost of CO₂ emission during its manufacture [4]. Alternative zonal isolation materials with similar or even better performance are indeed required to replace the dependency on Portland cement, especially for well construction and well abandonment.

Geopolymers, inorganic aluminosilicate polymers, are one of the potential green alternatives for OPC considering their production life cycle involving the utilization of solid-waste material [5,6]. The implementation of geopolymers in the oil and gas sector as

an isolation material has been under many challenges surrounding its applicability and efficiency in the field compared to the already established knowledge of OPC [7–9]. Solid precursors, of different origins, undergo a dissolution phase where components of highly complex minerals are disintegrated into smaller molecules, which form the basis of polymeric matrices composed of mainly Si–O–Al bonds [10]. Geopolymers require an activator (hardener), which acts as the dissolution medium of minerals where these hardeners can have a hydroxide nature such as potassium hydroxide (KOH) and sodium hydroxide (NaOH) or they can be of a silicate nature potassium/sodium silicate (K₂SiO₃/Na₂SiO₃) [11–14]. According to the work of different researchers Khalifeh et al. [15,16] and Duxson et al. [17], geopolymers poses properties that make them superior to OPC such as low chemical shrinkage, low permeability, high durability in corrosive mediums, and the ability of not being affected significantly by oil-based mud contaminants. The challenges evolve around admixtures to be utilized, temperature range efficiency, and hurdles in acquiring adequate properties. Although with these challenges, many efforts are being made by researchers to improve and formulate geopolymer mix designs into adequately applicable material in the field [18,19]. To make the geopolymers viable and sustainable for the industry, researchers should narrow the number of mix designs and diversity of geopolymer types based on the precursors used. This can facilitate mass production and reproducibility of the technology.

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Table 1 Solid precursor composition of mix designs (wt%)

Mix design	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	TiO ₂	MnO	SrO	BaO	S ²⁻	LOI	Total
A	62.01	9.84	0.42	16.74	5.73	1.76	1.79	1.01	0.001	0.001	0	0.52	0.13	100
B	62.51	9.92	0.43	16.06	5.78	1.78	1.81	1.02	0.01	0.01	0	0.58	0.13	100
C	69.75	10.02	0.58	10.21	3.72	2.33	2.41	0.67	0.01	0.01	0.01	0.37	0.2	100

It is well known that temperature affects the development of cementitious material significantly from rheology and workability to mechanical properties [20–24]. The development of geopolymer to have a working range of various temperatures can be of some challenge especially considering its ability to harden and form a solid material. In other words, one of the main remaining technical gaps for the commercialization of geopolymers is the development of few consistent mix designs that can be used in different temperature ranges. However, the proper utilization of the Ca²⁺ content in solid precursors can be a turning point to ensure strength development at low temperatures [25].

In this study, three different mix designs, i.e., three classes of a granite-based geopolymer are proposed considering the applicability of the geopolymer in well cementing and well abandonment operations using granite as a main solid precursor source. Calcium oxide (CaO) was introduced to mix designs to compensate for low reaction rates at low temperatures (<50 °C). Different classes have been assigned to different circulation temperatures ranging from 5 °C to 60 °C. The roles of operating temperature and granite-based composition are highlighted by studying short-term performance such as workability and setting time, rheology, uniaxial compressive strength (UCS), and X-ray diffraction measurements.

Materials and Methods

Mix Designs. A granite-based geopolymer solid precursor mainly composed of granite was used as the main solid phase in this study. One of the most influential factors is the Si and Al content considering the influence of Ca content, which plays an important role specifically at lower temperatures, thus CaO (purity >90%) was introduced to the mix designs at low temperatures [26]. In this work, CaO was added to the solid precursor and mixed with the dry components prior to mixing the slurry. This was conducted with mix designs A and B explicitly. Three mix designs were developed to tackle preliminary challenges of neat slurry where a range of temperature was considered for mix designs. Mix designs were labeled as A, B, and C where the first and second have quite close composition, except Ca content which goes higher as the working temperature decreases, and the third is studied for elevated temperatures. Different components have been introduced to tune the chemical composition to suite specified ranges of temperature and pressure. The composition maintained a mixture of granite, blast furnace slag, and microsilica throughout the three mix designs. Each of these components was proportionally mixed to give specific compositions suitable for the allocated temperature ranges. The chemical composition of each mix design is presented in Table 1.

The mix designs presented have been inspired by previous work conducted on granite-based geopolymers. Mix designs with the purpose of operating at low temperature applications where inspired from the work of Agista et al. [27] where it was foreseen the efficiency of KOH solutions in geopolymer slurry, especially at low temperature. CaO, added to the dry blend as specific weight percent of the solid, was used with the efforts to accomplish early mechanical properties and ensuring setting of the slurry at the recommended operational window [28,29]. In addition, the use of CaO was combined with KOH solutions rather than potassium silicate (K₂OSiO₂) solutions to avoid the formation of coagulants that may occur while using silicate solutions [30]. On the other hand,

mix design C has been inspired from the work of Chamssine et al. [18] where no CaO has been added to the system since it is well known that at elevated temperatures neat slurries achieve setting and mechanical properties can be measured easily. In addition, temperature highly influences geopolymer chemistry which assists in the formation of a firm, dense, and bulk structure [31–33]. Utilization of K₂SiO₃ instead of Na₂SiO₃ is due to lower viscosity and prolonged pumpability of the potassium silicate system. Slurry preparation was conducted using API 10B-2 recommended practices [34]. Mix design components are presented in Table 2.

Test Conditions. The temperature range used to test different mix designs was from 5 °C to 60 °C of bottomhole circulation temperature (BHCT). Temperatures were divided among the three mix designs as follows: mix design A was tested from 5 °C to 20 °C with a 5 °C increment between each test; mix design B was tested from 25 °C to 40 °C; mix design C was tested at 50 °C and 60 °C. Conditions of curing and testing are mentioned in Table 3. It must be noted that curing was done at the same mentioned temperatures since from field experience there is a risk of over cooling and uncertainty in reading downhole temperature, as the worst-case scenarios, which indicate BHCT and bottomhole static temperature are similar. This decision is justified when knowing the strength development of geopolymers is a strong function of temperature and wrong temperature selection can either result in flash setting or delayed setting. Such approach has been utilized in a study by Pernites et al. [35] on cement.

Workability. An atmospheric consistometer was used to evaluate the workability of mix designs at different working temperatures. All operations were handled following API 10B-2

Table 2 Mix design composition (wt%)

Mix design	Slurry design (wt%)					Solid/Liquid ratio
	Density (SG)	Solid	CaO	KOH (4 M)	K ₂ OSiO ₂	
A	1.88	69.50	3.0	30.50	—	2.28
B	1.88	69.30	1.0	30.70	—	2.26
C	1.98	66.70	—	—	33.30	2.00

Table 3 Operational conditions under study

Mix design	Pressure (bar)	Temperature (°C)
A	45	5
	55	10
	65	15
	80	20
B	95	25
	115	30
	145	40
C	175	50
	200	60

recommendations [34]. The selected ramp-up rate for temperatures above 25 °C selected to be 1 °C/min. The test was conducted once for each test's condition.

Conditioning. The atmospheric consistometer was used to condition samples for rheology and compressive strength sample preparations following API 10B-2 recommendations.

Rheology. The rheological properties of each geopolymer mix design were assessed using rotational flow tests at their designated temperatures, as outlined in Table 3. To evaluate the rheological characteristics of the geopolymer mix design at a specified temperature, a scientific rheometer was used. This instrument was chosen due to its ability to perform tests at low temperatures, down to 5 °C, which is not achievable with a standard API rotational viscometer. Rotational testing was conducted on each slurry under a controlled shear rate, where the shear stress was measured. Prior to testing, the samples were mixed and pre-conditioned using an atmospheric consistometer for 30 min at specified temperatures. The test was performed once for each material at different temperatures. The rheology test involved three different intervals, namely pre-shear, ramp-up, and ramp-down. The test began with pre-shearing at 100 1/s for 60 s, followed by ramp-up (0.01–511 1/s) and ramp-down (511 1/s to 0.01 1/s) stages.

Compressive Strength. The destructive method was used to measure the UCS of the samples, and a loading rate of 7 kN/min was applied which is in accordance with API 10TR-7 [36]. To conduct the test, each slurry was prepared using the same procedures as mentioned earlier, and pre-conditioned at a specified temperature before being poured into cylindrical plastic molds (5 cm in diameter and 10 cm in height). The molds were then placed inside pressurized autoclave cells and cured under controlled pressure and temperature for 1 and 7 days following on conditions mentioned in Table 3. Three samples were prepared for each mix design and the average was calculated.

X-ray Diffraction. The Bruker-AXS Micro-diffractometer D8 Advance was utilized to study crystallography of the precursors and observe phase changes, using $\text{CuK}\alpha$ radiation (40.0 kV, 25.0 mA) with a 2θ range from 5 deg to 92 deg, with a 1 deg/min step and a 0.010 deg increment. The samples were cured for 7 days at the maximum temperature limit of their respective mix designs. A, B, and C were cured at 20 °C, 40 °C, and 60 °C, respectively. Prior to testing, the cured samples were manually ground and dried in a vacuum oven at room temperature for 24 h.

Results and Discussion

X-ray Diffraction. Crystallography patterns of the solid precursor and cured samples of mix designs A, B, and C are presented in Figs. 1 and 2, respectively. The main phases recorded in both, the solid precursor, and cured samples, are quartz (Qz), albite (Alb), microcline (Mic), and exclusively a phase belonging to illite (IL) in the solid precursor part. The main change observed was the consumption of the IL after reacting and curing the solid precursor. This consumption can be due to the IL's tendency to dissolve in high alkaline environments [37]. The high concentration of Qz can be attributed to the presence of granite as the main precursor, a material rich of Si and Al species. It can be observed that the Qz concentration has decreased in mix design B indicating higher consumption of Si throughout the reaction in the allocated curing period. Seeing that the presence of Alb and Mic was detected, this can be an indicator of zeolite formation specifically since the material is in a K-activated system which favors the formation of zeolite phases [38]. This as well can be an explanation of why highly crystalline patterns were obtained where crystallinity was higher than 60% in all samples. High crystallinity can lead to an increase in

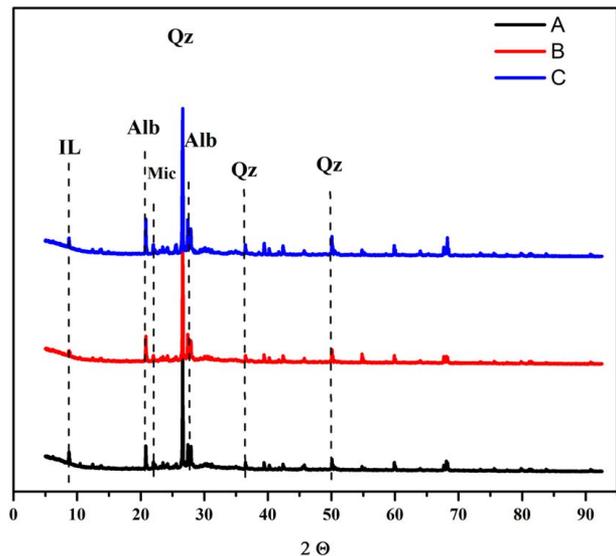


Fig. 1 XRD patterns for dry blend of mix designs A, B, and C

strength development however under thermal curing this can inhibit the formation of crystalline zeolites which counteracts strength development by applying inner stress or local destruction to the geopolymer's matrix [39]. These results clearly indicate the effect of temperature on reaction rates and the need to consider the balance between temperature and composition, since neither elevated temperature nor higher Ca content can aid unless it is formulated in a balanced manner to serve the type of application and ensure favorable kinetics of reaction.

Workability. The first field parameter to discuss is workability of the different mix designs under different temperatures. Workability measurements of different mix designs at different temperatures are presented in Fig. 3. Each mix design has been tested at the specific assigned range since it is foreseen from previous studies that the use of more Ca species at lower temperatures can be highly beneficial for the workability of the slurry and the opposite at higher temperatures [18,25,27]. It can be observed how temperature increase has an accelerating effect on the initial and final setting

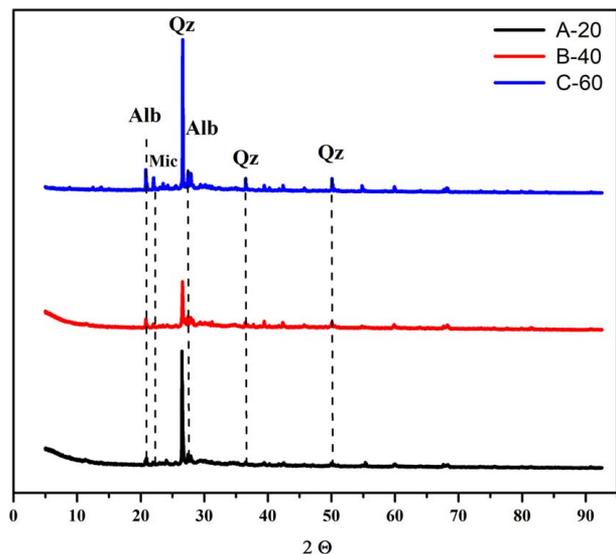


Fig. 2 XRD patterns for mix designs A, B, and C cured for seven days at 20, 40, and 60 °C, respectively

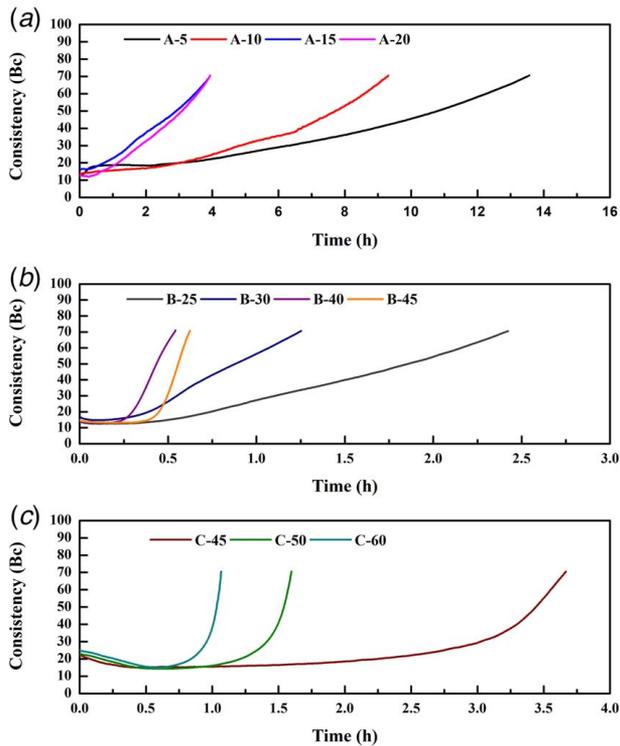


Fig. 3 Workability measurement of mix designs at different temperatures: (a) mix design A, (b) mix design B, and (c) mix design C. Each curve tested at the specific temperature indicated.

time of geopolymer mix designs, especially mix design A at 20 °C and at 5 °C. Temperature can facilitate the setting of geopolymer material which in turn can accelerate the development of hard structure [40,41]. In addition, to properly understand how different materials behave at the intermediate temperature zones, different mix designs were tested at their temperature limitations. Focusing on mix design B, the temperature limitation can be observed at 45 °C, an intermediate point between mix design B and mix design C. It must be noted that due to the rapid setting observed by mix design B at 45 °C, it was decided to switch to K_2OSiO_2 solution as it has proven its efficiency under elevated temperature conditions [18]. This shift to K_2OSiO_2 solution and a significant decrease in Ca content in mix design C created a synergy in the slurry allowing the prolongation and safe handling of slurry at temperatures exceeding the 45 °C temperature limit to go up to 60 °C.

Throughout this study, the criteria of assigning working temperatures for different mix designs depended on mainly Ca content in mix designs. Duxson et al. [42] highlighted the properties of low Ca geopolymer at low temperatures where hardening time is quite slow compared to samples cured at elevated temperature. Nath and Sarker [43] examined the efficiency of OPC for fly ash class F geopolymers cured at ambient conditions. They concluded that the presence of OPC not only accelerated the reaction of geopolymerization but also influenced the compressive strength of the material; they attributed this increase to OPC addition as a Ca source for the reaction which in turn yielded C-S-H phases that was able to accelerate the polycondensation phase of the reaction. The proper utilization of the Ca^{2+} content in solid precursors can be a turning point to ensure strength development at low temperatures [25]. Thus, the tuning of Ca content can have a major effect on slurry properties at low temperatures as demonstrated in mix design A at 5 °C, which was handled at relatively low operational temperature but still managed to harden due to the higher Ca content in the dry blend.

Rheology. The rheology tests through ramp-up and ramp-down tests were performed, and the ramp-down test result is presented in Fig. 4. Ramp-down test is selected due to its representation of dynamic condition of the slurry. The effect of the temperature is noticeable in the different mix designs. Higher temperatures will induce higher kinetic energy, increasing the molecule vibration of the carrier fluid hence reducing the slurry's viscosity [27]. This behavior can be seen in the mix design A which tested at 5–20 °C. On the other hand, the viscosity result for mix design B shows the opposite trend with the increasing temperature. Increasing temperature will result in increased viscosity of both mix designs. At this stage, the slurry is in the acceleration phase in which increasing temperature will fasten the geopolymerization reaction leading to hardening of slurry. Moreover, the viscosities of mix design B at 40 °C and mix design C at 60 °C were not able to be measured due to fast hardening at that specific temperature. This finding is consistent with the consistency test results presented in Fig. 3, indicating that both slurries were pumpable for less than 1 h. The outcome of the study revealed that mix designs A and B are unsuitable for use at elevated temperatures as they are significantly affected by gelation and hardening due to the presence of Ca. Similarly, mix design C also experienced fast setting at temperatures above 60 °C and could not perform well for low temperatures as shown in Fig. 3.

Compressive Strength. The performance of different mix designs at their designated temperature ranges is presented in Fig. 5. Tests for each sample were repeated for three times and

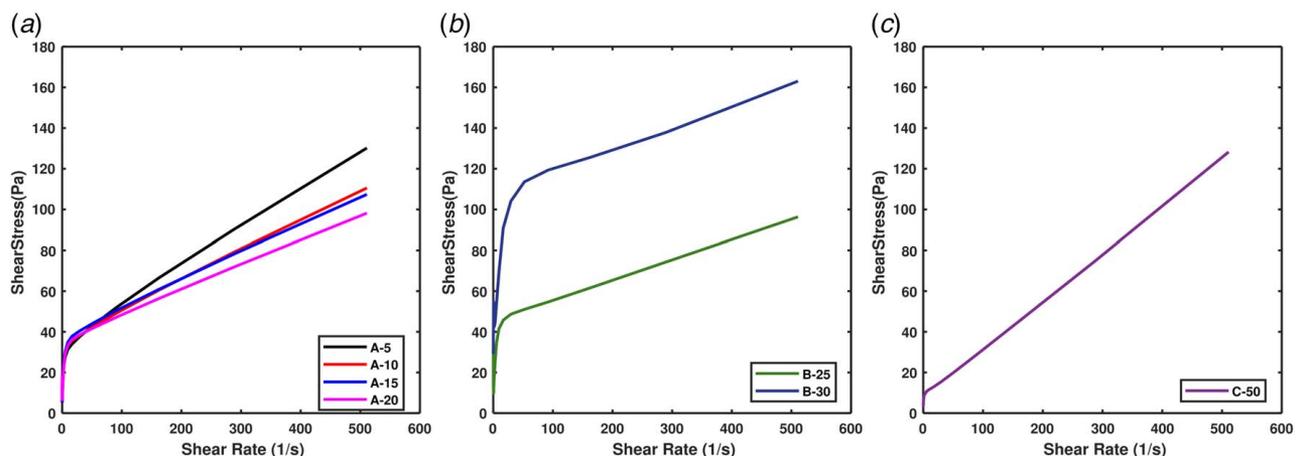


Fig. 4 Rotational flow (ramp-down) measurement of mix designs: (a) mix design A, (b) mix design B, and (c) mix design C

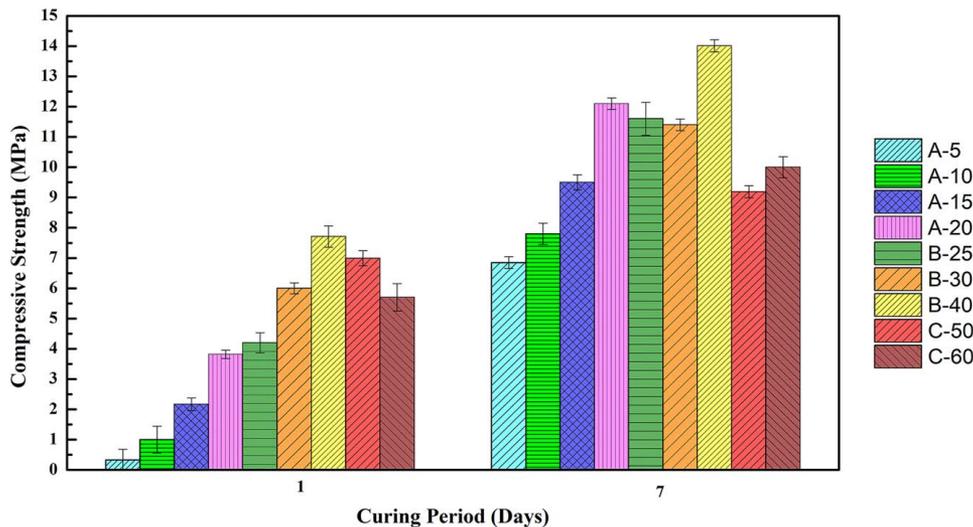


Fig. 5 Average compressive strength of mix designs cured at allocated temperatures

the average is presented with the standard error bars connected to each test. The curing conditions under different operational temperatures have caused a significant change in the materials' ability to withstand mechanical load. Temperature has a direct impact on the strength development of material, and it can be correlated to OPC at elevated temperatures [44]. However, the material's composition, specifically Ca content, contributes hugely to the reaction rate of the mix design and eventually its strength [29]. The increase in Ca content can promote the formation of minor C-S-H phases aiding the matrix's strength development phase [45]. It can be observed that mix design A at 5 °C yielded the lowest compressive strength after one-day of curing compared to samples cured at higher temperatures despite having a high Ca content. These results are expected due to the increase in temperature. However, interestingly observing mix design B at 25 °C it starts to show that although with a lower Ca content in comparison to mix design A samples, compressive strength keeps on increasing with increasing temperature which highlights the significant effect temperature can make on the slurry reaction, hence the effect on compressive strength. In addition, mix design B at 40 °C had the highest strength in comparison to all other samples which seems that the reaction at 40 °C is more favorable kinetically where more intermolecular matrices are developing [46]. However, more investigation is required to observe kinetic behavior within every mix design individually at allocated operational temperatures. Moreover, it was observed that mix designs C at 50 °C and at 60 °C have measured quite close to each other. Although being cured at elevated temperatures, the temperature effect could not compensate for the missing Ca content in comparison to mix design B's composition. This leads up to a point of interest addressing the firm balance to be made between temperature effect and Ca content in every mix design, which asks for deeper understanding and development of a correlation between both parameters at a kinetic level.

Conclusion

Three geopolymers mixed designs were devised to handle different sections of well cementing, each having a unique composition. Different tests were performed at a range from 5 °C to 60 °C considering applicability of each mix design at a specific temperature range. The results for each mix design have been treated individually where their behavior in different conditions, with different Ca content, was the focus point in this study. Based on X-ray diffraction (XRD) readings, the mix designs had consistent and similar mineral detection which indicates that Ca content, due to its minor presence in comparison to other elements such as Si and Al, was not highly visible. However, mix design B had an

interesting Qz phase post curing which indicates favored reaction kinetics for the reaction at 40 °C. Workability indicated the benefit of Ca content at lower temperatures and how temperature was one of the most sensitive parameters determining the performance of geopolymer material and affecting the selection of composition for each mix design. Furthermore, compressive strength increased with increasing curing temperature where mix design B achieved the highest performance at 40 °C. This high performance can be attributed to favorable reaction conditions which were supported by XRD patterns of cured samples where high consumption of Qz can be observed in mix design B. The mean of developing geopolymer material for oil and gas application should consider a wide range of parameters that can be suitable for producing a successful material where composition and temperature range can highly impact the rate of reaction and thus the efficiency in developing strong matrices that can withstand external parameters. Further recommendations will be to aim for developing chemical admixtures, in different capacities, that would lift the material's properties and engineer compositions capable of successful implementation into field usage. Plus, the Ca content in geopolymer material must be tuned and considered for implementation in field applications due to its beneficial effect on the bulk matrix, specifically in ensuring setting of slurry and achieving minimum acceptable compressive strength post-setting.

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Conflict of Interest

There are no conflicts of interest. This article does not include research in which human participants were involved. Informed consent was not applicable. This article does not include any research in which animal participants were involved.

Data Availability Statement

The authors attest that all data for this study are included in the paper.

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