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PRODUCTION AND CHARACTERIZATION OF GEOPOLYMERS FROM DIATOMACEOUS EARTH

Abstract. The world has been facing an increase in the average surface temperature of the earth due to the high release of carbon dioxide into the atmosphere. One of the main responsible for this release of CO₂ is the manufacture of Portland Cement, associated with an emission of carbon dioxide of around 7% of the world's total emissions. In this way, there is a need to find alternatives to Portland cement. This work aims to contribute to this endeavor, proposing the application of a solid waste used as a wine filtration agent in the wine industry, diatomaceous earth, containing high amounts of silicon, to produce geopolymers. A geopolymer is an inorganic polymer produced with an aluminosilicate precursor reacted with an alkaline solution that has been studied as an alternative to cement. Diatomaceous earth and alumina were employed in this work as aluminosilicate sources for the geopolymer precursor, while sodium hydroxide and sodium silicate were used as the alkaline solution. The production process involved mixing all these raw materials of the geopolymers to create a fresh geopolymer. Following production, the geopolymers were characterized using XRD, FTIR, SEM-EDS, and pore property analysis. The results revealed that the most favorable geopolymer was produced considering a NaOH concentration of 10 M and a Si/Al ratio of 3,5. This combination resulted in a higher proportion of geopolymer phase, consequently improving mechanical properties.

Keywords: diatomaceous earth; geopolymers; portland substitute.



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Introduction. Since the Industrial Revolution, the world has faced consequences of industrial expansion and fossil fuel combustion, such as the accumulation of greenhouse gases (GHGs) in the atmosphere [1]. The

concentration of carbon dioxide (CO₂) has significantly increased in the atmosphere by 30% since 1950 [2], caused most likely by human activities, such as forestation for agriculture and forestry [2], industrialization [3], transportation [4] and the building sector, which appears among the most responsible for this situation, accounting for almost 38% of the total emissions of CO₂ [5,6].

Portland Cement (PC)-based materials are the second most used substances in the world after water. Buildings, bridges, and roads would be impossible without these materials. Because of this, cement is the most extensively manufactured product worldwide, with a global annual production of about 4 billion tons. This manufacturing process consumes a massive amount of energy and raw materials. Besides, in 2021, the cement industry emitted nearly 2,9 billion tons of carbon dioxide, which is more than 7% of the global carbon emissions [7-12].

Some of the alternatives to PC are (i) the partial replacement of this material, (ii) the production of alternative cement with low-energy, e.g., calcium sulfoaluminates, or (iii) the formulation of new binders, such as geopolymeric materials. Geopolymer is an alkali-activated binder made by alkali activation of aluminosilicate source materials such as fly ash (FA), metakaolin (MK), and Diatomaceous Earth (DE), among others. The emission of CO₂ by geopolymers is said to be 80% lower compared to the production of ordinary Portland cement [8,13].

Thus, this study aims to investigate the application of Spent Diatomaceous Earth (SDE), a solid waste generated from beverage purification, such as wine, as a source of aluminosilicate for the production of geopolymers.

Materials and methods *Chemicals and apparatus.* The following reactants were used in this study: spent diatomaceous earth provided by Caves Campelo, aluminum oxide (Al₂O₃ – 99,7 % Thermo Scientific), sodium hydroxide (NaOH, 98.73%, Fisher Chemical, U.K.), sodium silicate solution (H₁₀Na₂O₈Si—Na₂O = 10.6% and SiO₂ = 26.5%, Fisher Scientific), standard sand (EN 196-1—Societe Nouvelle du Littoral), and Portland Cement (32,5N—Secil). Potassium bromide (>99 wt.%, Sigma Aldrich) was used to prepare pellets for Fourier-transform infrared analysis.

Geopolymer production. The geopolymer composition was chosen after the characterization of the SDE used as a precursor, identified elsewhere [14]. Finally, the influence of the concentration of alkaline solution and the Si/Al ratio were evaluated. The produced geopolymers used alkaline solutions based on sodium hydroxide of 10 and 12 M and sodium silicate (SS). Si/Al ratios of 2,5 and 3,5 were chosen to follow the literature. The samples were cured for four days in oven at 40 °C and then at room temperature. Finally, the geopolymers were identified as GP1 (NaOH 10 M and Si/Al 2,5), GP2 (NaOH 10 M and Si/Al 3,5), GP3 (NaOH 12 M and Si/Al 2,5), and GP4 (NaOH 12 M and Si/Al 3,5).

Characterization techniques. The characterization techniques, such as inductively coupled plasma-optical emission spectrometry (ICP-OES), and X-ray diffraction (XRD), were performed as reported in previous studies [14]. Fourier-transform infrared spectroscopy (FT-IR) was conducted on pellets of samples prepared with KBr using a Perkin Elmer FT-IR spectrophotometer UATR Two to study the functionalities present in the samples. N₂ adsorption-desorption isotherms at 77 K were obtained using a Quantachrome NOVATOUGH LX [4] adsorption analyzer to determine the textural properties of the nanoparticles. Scanning Electron Microscopy with energy-dispersive X-ray Spectroscopy (SEM/EDS) analysis is a highly versatile tool that can examine and analyze the microstructural

properties of solid objects. The samples analyzed in this study were collected in powder form and were examined at scales of 100, 20, and 10 μm .

These characterization techniques provided valuable insights into the geopolymer's structural and chemical properties.

Research results and discussion. *Geopolymer characterization.* The XRD analysis of geopolymers in contrast to diatomaceous earth reveals distinct crystalline peaks. Fig. 1 illustrates the diffractogram of diatomaceous earth alongside geopolymers formulated with various compositions.

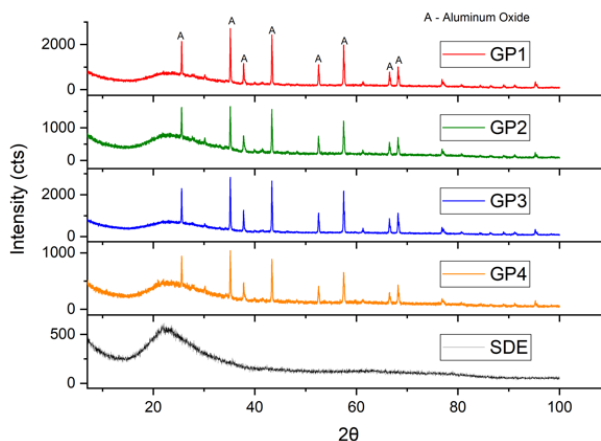


Fig. 1. XRD results for GPs

The major peaks are associated with the aluminum oxide phase at $25,57^\circ$, $35,14^\circ$, $37,77^\circ$, $43,35^\circ$, $52,54^\circ$, $57,48^\circ$, $66,51^\circ$, and $68,20^\circ$ in all four GPs [15,16]. These peaks originate from the alumina added to the precursor, some of which are repeated in the diffractogram of the geopolymers. The alumina pattern is shown in Fig. 2, and it can be seen that the resulting geopolymers do not have all the peaks present in the raw material, as those at $41,61^\circ$, $46,18^\circ$, $59,77^\circ$, $70,36^\circ$, $74,27^\circ$, $85,19^\circ$ and $90,66^\circ$, confirming a certain degree of consumption of this alumina in the geopolymerization reaction [17].

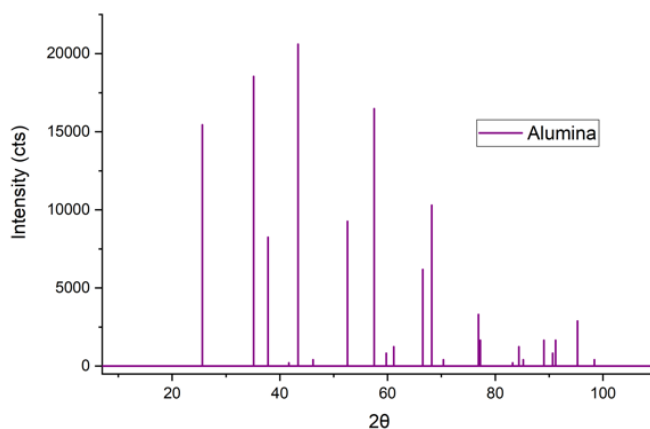


Fig. 2. XRD for alumina pattern

However, the prominent peaks persisted, although with significantly reduced intensity, indicating the presence of partially unreacted crystalline material. When comparing geopolymers based on their Si/Al ratio, it can be observed that GP1 and GP3 exhibit more pronounced peaks of aluminum oxide compared to GP2 and GP4. This can be attributed to the fact that GP1 and GP3 have Si/Al ratios of 2,5, which indicates a higher amount of alumina present in the geopolymer, in contrast to GP2 and GP4, which have Si/Al ratios of 3,5. The higher alumina concentration in GP1 and GP3 likely contributes to the observed differences in the peaks of aluminum oxide between these materials and GP2 and GP4.

The geopolymerization process was described by four mechanisms. They are influenced by the amount of alkaline solution and its concentration once the amount of OH^- and Na^+ needs to be sufficient to dissolve the aluminum from the precursor and to balance the negative charge of the $\text{Al}(\text{OH})^-$, respectively. If there is more aluminum than the maximum for the reaction, less NASH gel is generated, and more unreacted Al is presented [18]. This is a possible explanation why the amorphous halos of GP1 and GP3 are less intense than GP2. The first and the third samples have a Si/Al ratio of 2,5, so the amount of aluminum oxide powder added to the precursor was higher, causing an excess of Al. This also goes according to the conclusion of unreacted aluminum described by the intensity of the crystalline peaks.

Fig. 3 presents the FTIR results of SDE and all the geopolymers produced. The peak with the highest intensity corresponding to the adsorption band at 1020 cm^{-1} can be related to the vibration of the asymmetric stretching of the Si-O-Si bond, which can be seen in SDE and GP samples.

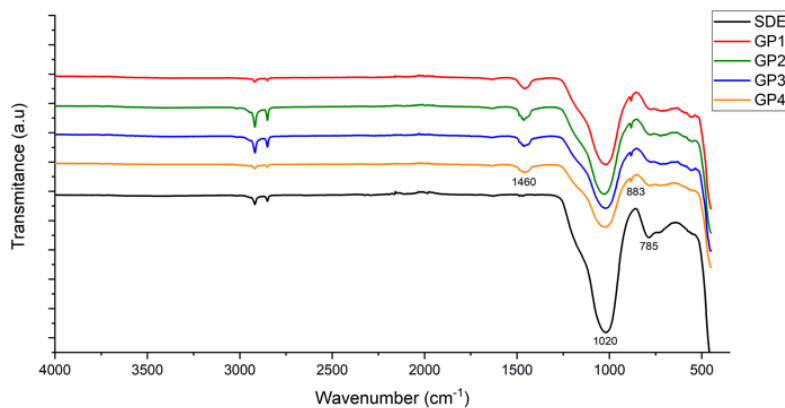


Fig. 3. FTIR results for GPs

Studies in the literature examining the correlation between the Si/Al ratio caused by geopolymerization and the frequency (cm^{-1}) at which peaks occur concluded that an increase in the quantity of Al components results in a gradual shift of the peaks generated through geopolymerization to lower frequencies [19]. However, the results of this work show that the peak with highest intensity observed in GPs did not exhibit any significant dislocation. This observation can be attributed to the findings of the XRD analysis, which revealed that not all of the alumina was involved in the geopolymerization process. As a result, there was a deficiency in forming a strong Si-O-Al bond, which was expected to occur in frequencies close to 990 cm^{-1} , according to the literature.

In the FTIR analysis of geopolymer samples, the emergence of new bands near frequencies of 883 and 1460 cm^{-1} can be observed when compared to the raw material. The band at 1460 cm^{-1} corresponds to the antisymmetric vibrations of CO_2^3 , indicating the presence of sodium carbonate in the geopolymer's structure. Additionally, the absorption band at 883 cm^{-1} is associated with the Si-O-Al stretching vibration following XRD analysis, a specific part of alumina reacted in the geopolymerization process.

Fig. 4 presents the SEM images of the produced GPs. GP1 and GP3 samples, which have the same Si/Al ratio of 2,5, exhibited the irregular shapes and sizes of a flocculent morphology covered with fine powder spheres [20]. These attached spheres can be due to the higher quantity of alumina powder added to these samples to achieve the desired Si/Al ratio [21], but they didn't react, reinforcing the XRD analysis. These images also exhibited smaller pores and more compact structures.

Meanwhile, GP2 and GP4 samples presented mainly a flocculent morphology with more prominent pores and a less compact structure. Needle-like structures were also detected in GP4. This type of structure is similar to zeolitic-fibrous phases, which can propose a small amount of a poor crystalline phase of zeolite in the sample [20,22], going according to what was analyzed in XRD.

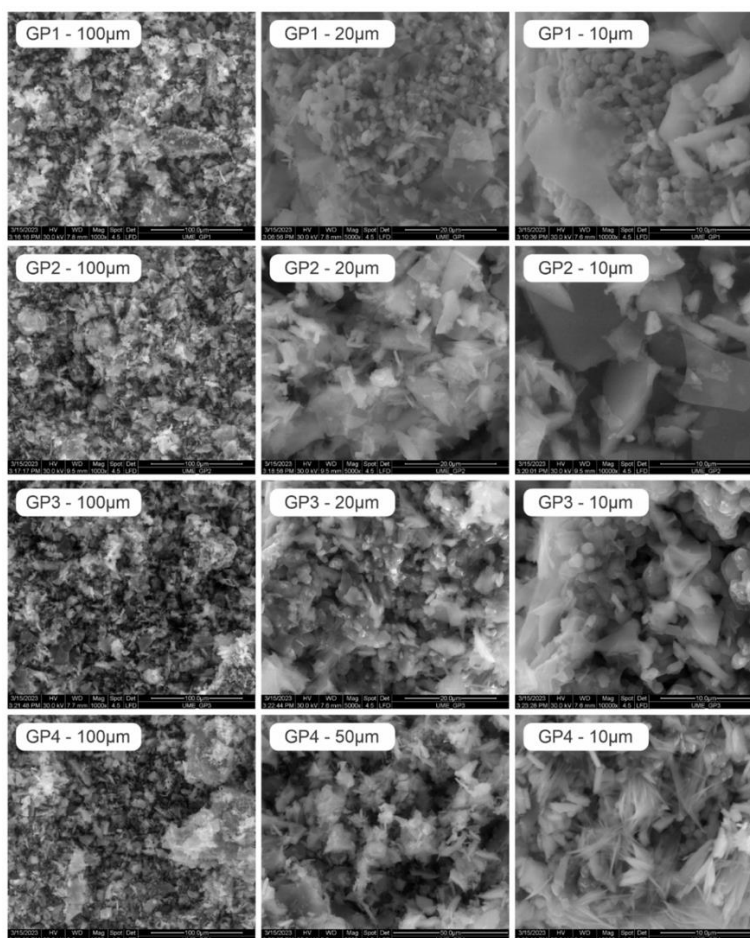


Fig. 4. SEM Images of GPs

The elemental analysis of the geopolymers by energy dispersion X-ray, as depicted in F.

Fig. 5, showed the presence of silicon, aluminum, and oxygen, which was consistent with the expected results of a polysialate material [23]. Compared to the EDS result of SDE, it was observed that the intensity of Si, Al, O, and Na had increased. The presence of sodium was intensified using NaOH solution, which served as the alkaline solution to prepare geopolymers. The silicon and aluminum content increase can be attributed to adding SS solution and alumina powder, respectively. These results align with the findings of the FTIR analysis, which indicated the formation of a Si-O-Al bond at the absorption band of 883 cm^{-1} .

The graphics also indicate that the intensity of the silicon peak is higher in GP2 and GP4 compared to GP1 and GP3, which confirms the higher Si/Al ratio desired in GP2 and GP4 formulations. Furthermore, this composition helps assume that there was a NASH formation [22], as described in the XRD analysis.

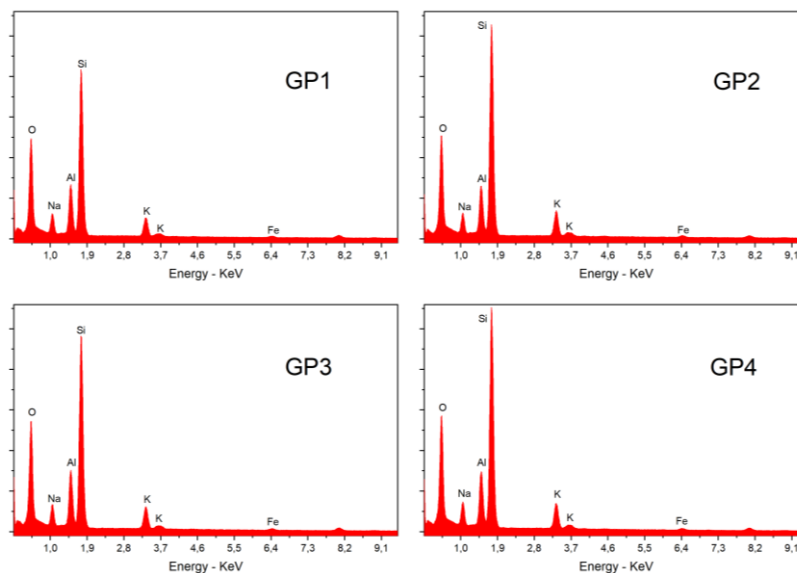


Fig. 5. EDS results for GPs

The N_2 adsorption-desorption isotherms of GPs are illustrated in

Fig.. According to the classification of physisorption isotherms suggested by IUPAC [24], it should be noted that GP1, GP3, and GP4 seem to exhibit an isotherm of type III, typical of materials with low porosity [25], following the exact characteristics of the precursor SDE.

GP2, as described before, is the sample with more NASH or geopolymeric phase, so this analysis follows the literature, which indicates that the more geopolymeric phase, the more the material will have mesoporous characteristics, once this is the most common type of pores in the geopolymer gel [26]. GP2, however, presents an isotherm of type IV, indicating that the sample may be a mesoporous material.

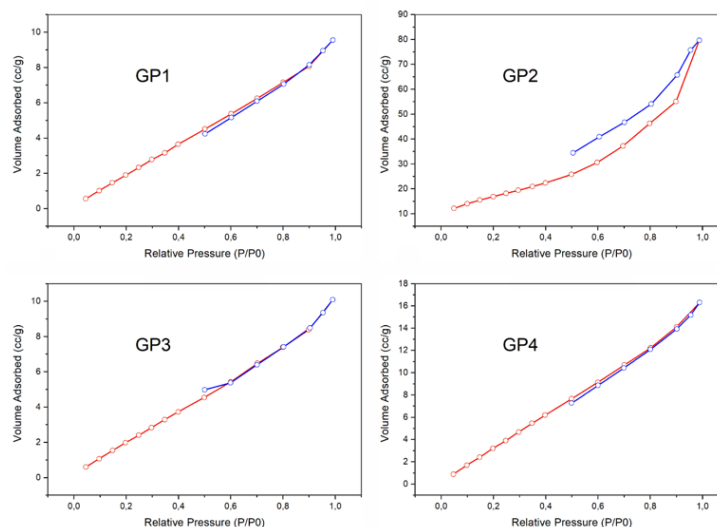


Fig. 6. Geopolymers Isotherms

Fig. 6 also shows the difference between the volume adsorbed by GP2 and the other GPs, indicating an elevated specific surface area on the sample, as shown in Table 1 through BET methodology. GP2 and GP4 present a specific surface area more extensive than GP1 and GP3, especially GP2, with an S_{BET} four times higher than the SDE sample. It brings a positive property to the sample once a higher surface area represents higher reactivity, resulting in higher compressive strength [27]. Also, GP1 and GP3 may have a small S_{BET} due to the particles of the unreacted aluminum shown in SEM images, which can occupy space on the surface, reducing its area and the material's reactivity.

Table 1

Pore Structure Parameters of SDE and GPs

Sample	Specific surface area BET (m^2/g)	Pore Volume (cm^3/g)	Average pore radius (nm)
SDE	15	0.0156	1.81
GP1	12	0.0127	1.81
GP2	60	0.1104	2.30
GP3	12	0.0135	1.81
GP4	23	0.0219	1.81

Conclusion. Based on the comprehensive analysis, it can be concluded that SDE is a viable aluminosilicate source for geopolymer precursors. This demonstrates that it is possible to produce geopolymers from SDE. Remarkably, being a solid waste, its use significantly reduces the need for extracting natural resources. Chemical analysis revealed higher geopolymerization in GP2, highlighting the material's potential. Moreover, the incorporation of this geopolymer as a partial or complete binder in mortar formulations, is promising to obtain materials with the potential for future applications in civil construction. Further enhancements can be achieved by combining it with other aluminosilicates.

This approach promises a more sustainable trajectory for the construction industry, promoting sustainable development and circular economy principles.

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ДИАТОМДЫ ЖЕРДЕН ГЕОПОЛИМЕРЛЕРДІ АЛУ ЖӘНЕ СИПАТТАУ

Аңдатпа. Әлем атмосфераға көмірқышқыл газының көп бөлінуіне байланысты жер бетінің орташа температурасының жоғарылауына тап болды. Бұл СО₂ шығарындыларының негізгі себептерінің бірі-әлемдегі жалпы шығарындылардың шамамен 7 % құрайтын көмірқышқыл газының шығарындыларымен байланысты

портландцемент өндірісі. Осылайша, портландцементке балама іздеу қажеттігі туындады. Бұл жұмыс шарап өнеркәсібінде шарапты сүзу құралы ретінде пайдаланылатын қатты қалдықтарды – құрамында геополимерлерді алу үшін қажет көп мөлшерде кремний бар, диатомды жерді пайдалануды ұсына отырып, осы бастамаға үлес қосуға бағытталған. Геополимер-цементке балама ретінде зерттелген сілтілі ерітіндімен әрекеттескен алюмосиликатты прекурсордан алынған бейорганикалық полимер. Бұл жұмыста геополимер прекурсорын алу үшін алюмосиликат көзі ретінде диатомды жер мен глинозем, ал сілтілі ерітінді ретінде натрий гидроксиді мен натрий силикаты пайдаланылды. Өндіріс процесі жаңа геополимер алу үшін геополимерлердің барлық шикізат компоненттерін араластыруды қамтыды. Өндірістен кейін геополимерлер рентгенография, FTIR, SEM-EDS және кеуек қасиеттерін талдау арқылы сипатталды. Нәтижелер ең оңтайлы геополимер NaOH 10 М концентрациясында және Si/Al 3,5 қатынасында алынғанын көрсетті. Бұл комбинация геополимер фазасының үлесінің артуына әкелді, сондықтан механикалық қасиеттерін жақсартты.

Тірек сөздер: диатомды жер, геополимерлер, портландцементті алмастырғыш.

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ПОЛУЧЕНИЕ И ХАРАКТЕРИСТИКА ГЕОПОЛИМЕРОВ ИЗ ДИАТОМОВОЙ ЗЕМЛИ

Аннотация. Мир столкнулся с повышением средней температуры поверхности Земли из-за большого выброса углекислого газа в атмосферу. Одной из основных причин такого выброса CO₂ является производство портландцемента, связанное с выбросами углекислого газа, составляющими около 7 % от общего объема выбросов в мире. Таким образом, существует необходимость в поиске альтернатив портландцементу. Данная работа направлена на то, чтобы внести свой вклад в это начинание, предложив использовать твердые отходы, используемые в винодельческой промышленности в качестве средства для фильтрации вина, - диатомовую землю, содержащую большое количество кремния, - для получения геополимеров. Геополимер - это неорганический полимер, полученный из алюмосиликатного предшественника, вступившего в реакцию со щелочным раствором, который был изучен в качестве альтернативы цементу. В данной работе в качестве источников алюмосиликата для получения прекурсора геополимера использовались диатомовая земля и глинозем, а в качестве щелочного раствора - гидроксид натрия и силикат натрия. Процесс производства включал в себя смешивание всех этих сырьевых компонентов геополимеров для получения свежего геополимера. После производства геополимеры были охарактеризованы с помощью рентгенографии, FTIR, SEM-EDS и анализа свойств пор. Результаты показали, что наиболее оптимальный геополимер был получен при концентрации NaOH 10 М и соотношении Si/Al 3,5. Такое сочетание привело к увеличению доли геополимерной фазы, что, следовательно, улучшило механические свойства.

Ключевые слова: диатомовая земля, геополимеры, заменитель портландцемента.