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Hybrid cements from mining tailings for possible uses in construction

Cementos híbridos a partir de relaves mineros para posibles usos en construcción

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KEYWORDS
Alkaline activated cements; geopolymers; hybrid cements; construction materials; mine tailings
Cementos activados alcalinamente; geopolímeros; cementos híbridos; materiales de construcción; relaves mineros.

ABSTRACT: The Department of Antioquia, Colombia, produces 47% of the mining tailings linked to national gold extraction. These residues, stored in dam-like deposits, contain toxic substances such as heavy metals, posing serious risks to the environment and public health. Due to their high silica and alumina content, these tailings could be repurposed as alternative cementitious materials. Hybrid cements, formed by combining Portland cement with alkaline-activated materials, offer a lower environmental impact and support circular economy practices. In this study, the potential use of Antioquia's mining tailings as precursors for hybrid cement production was evaluated. Sodium carbonate and sodium silicate were used as alkaline activators, and curing was performed at room temperature. The tailings' chemical composition showed suitable levels of silica and alumina for geopolymer synthesis. Concrete specimens were prepared using hybrid cement and three types of aggregates: sand, coarse tailings, and recycled polymer. After 28 days of curing, the highest compressive strength was 568.6 kN ± 6.1 with coarse tailings, followed by 550.3 kN ± 7.9 with sand, and 325.3 kN ± 7.5 with recycled polymer. These results demonstrate the feasibility of using mining residues in sustainable construction applications.
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RESUMEN: El Departamento de Antioquia, Colombia, genera el 47 % de los relaves mineros asociados a la extracción nacional de oro. Estos residuos, almacenados en depósitos tipo represa, contienen sustancias tóxicas como metales pesados, representando riesgos ambientales y de salud pública. Por su alto contenido de sílice y alúmina, pueden aprovecharse como materiales cementantes alternativos. Los cementos híbridos, que combinan cemento Portland con materiales activados alcalinamente, presentan menor impacto ambiental y favorecen la economía circular. En este estudio, se evaluó el uso de relaves mineros de Antioquia como precursores para cementos híbridos, empleando carbonato y silicato de sodio como activadores alcalinos y curado a temperatura ambiente. La caracterización química confirmó niveles adecuados de sílice y alúmina para la síntesis de geopolímeros. Se prepararon concretos con dicho cemento híbrido y tres tipos de agregados: arena, relave grueso y polímero reciclado. Tras 28 días de curado, las mayores resistencias a compresión fueron: 568.6 kN ± 6.1 con relave grueso, 550.3 kN ± 7.9 con arena, y 325.3 kN ± 7.5 con polímero reciclado. Los resultados respaldan la viabilidad de emplear estos residuos en aplicaciones constructivas sostenibles.

1. Introduction

The expansion of mining operations in Antioquia, Colombia, has resulted in a considerable accumulation of tailings, which, as a byproduct of this industry, present significant environmental and social challenges. The residues, which are primarily composed of unused minerals and residual chemical substances, have the potential to cause contamination of soils and bodies of water if they are not appropriately managed. Considering these circumstances, the pursuit of sustainable solutions for the management of these wastes has become imperative.

While the precise number of gold mines in Antioquia is not currently known, the Department's Secretary of Mines and the Mining Census of Antioquia estimated that there were approximately 2.025 mining operations by 2011. A significant proportion of these were dedicated to the extraction of gold. It is evident that this figure has been on the rise, consequently leading to an increase in the production of gold mining tailings.

In this context, mining residues represent a promising source of secondary raw materials for the production of alternative cements. This strategy not only offers an efficient approach to managing tailings but also helps reduce the environmental impact of the cement industry, one of the largest global emitters of carbon dioxide (CO_2). Among the most studied alternatives are alkali-activated cements, such as geopolymers and hybrid cements, which use silica- and alumina-rich materials like mining waste as precursors [1], [2].

However, the use of mining residues must take into account the potential presence of toxic substances, such as heavy metals (Pb, Hg, Cd, As, Cr, etc.), sulfides, respirable crystalline silica, and natural radionuclides. Prolonged exposure to these elements may lead to adverse effects on human health, including systemic toxicity, respiratory diseases, and in some cases, carcinogenic risks. Therefore, it is essential to implement inertization, encapsulation, and monitoring processes to ensure these compounds do not leach into the environment or impact public health [3], [4] The Alkaline Activated Cements (AAC) or geopolymers, are a type of cementitious material with mechanical and chemical properties comparable to those of Ordinary Portland Cement (OPC), but with a significantly lower environmental footprint. AACs are obtained by alkaline activation of materials rich in silica and alumina, such as mine tailings, allowing their transformation into high-value-added products. In addition to offering a solution to waste disposal, the geopolymers reduce CO_2 emissions associated with conventional cement production.

From a chemical perspective, the most crucial aspect in the formation of AAC is the silica and reactive alumina content in the initial aluminosilicate, as silicon serves as the primary component of the structural skeleton of the reaction products formed during the alkaline activation of the material. The geopolymerization process involves a chemical reaction between aluminosilicate materials and highly alkaline solutions such as silicates, hydroxides or carbonates of alkali metals. This reaction produces amorphous to semi-crystalline three-dimensional polymeric structures comprising siloxo-sialate bonds (Si-O-Si-O-Si-O-Al) [5]. For a visual representation of this phenomenon, see **Figure 1**.

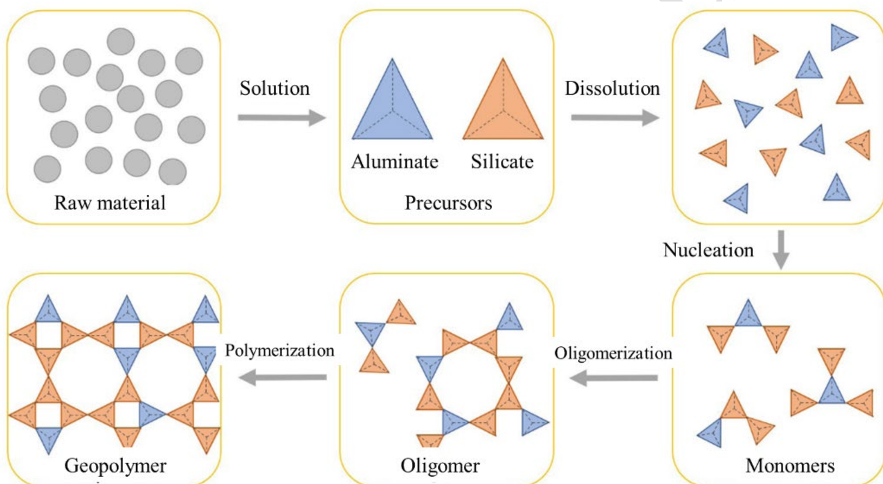

Figure 1. Geopolymerization process (Adapted from [6]).

Furthermore, it has been proven that geopolymerization can encapsulate dangerous or harmful elements from the mineral beneficiation process. This represents a major advantage for its application in construction.

On the other hand, alkali-activated hybrid cement (AAHC) -is a mixture of an alkaline activated cement plus OPC- has also been investigated as an option for partial replacement of OPC [7-11]. These cements are OPC-containing binders, and therefore, have approximately 20% of CaO, SiO_2, and Al_2O_3, producing gels whose formation varies according to the process conditions. Among the most studied AAHC systems are mixtures between OPC and blast furnace slag, metallurgical phosphate slag, and fly ash [10]. It should be noted that research has shown that the presence of reactive calcium in the clinker has allowed the curing of these mixtures without the need to increase the temperature, that is, at room temperature [12-15].

It is important to highlight that hybrid cements have made substantial progress in research, demonstrating favorable outcomes in terms of mechanical strength, durability, chemical resistance, and performance in aggressive environments (such as exposure to sulfates, acids, and chlorides) [16]. The controlled combination of OPC and alkaline precursors enables the beneficial hydration of clinker while simultaneously activating the geochemical reactions of aluminosilicate materials. One of the most significant advances is their ability to cure at room temperature, which enhances their practical applicability [17], [18]

Nonetheless, the lack of consolidated international standards regulating their commercial use remains a challenge. There is a pressing need for formulation standardization and further research aimed at homogenizing raw materials to ensure consistent performance. These steps are essential to promote broader adoption within the construction industry [19], [20]

In view of this problem, there is a need to explore sustainable solutions that allow the valorization of these wastes. A promising alternative is the use of mine tailings as raw material to produce geopolymers. In light of the aforementioned considerations, this article seeks to examine the potential of mining tailings from an area of Antioquia-Colombia for the production of alkali-activated hybrid cement. This process involves the use of sodium carbonate and sodium silicate as alkaline activators, with a curing procedure conducted at room temperature. Furthermore, the concretes produced with this hybrid cement were evaluated using sand, coarse tailings, and polymer as aggregates. The compressive strength was evaluated at 7, 14, and 28 days of curing. It is anticipated that this preliminary study will contribute to the advancement of knowledge regarding the development of a sustainable alternative for the mining and construction sectors in the region. This approach is aligned with the principles of the circular economy and the reduction of environmental impact.

2. Materials and methods

2.1. Physical preparation of samples

The mining tailings (MT) used to prepare Alkali-Activated Hybrid Cement with Mining Tailing (AAHC-MT) were sourced from one mining site in Antioquia, Colombia. In order to enhance the material surface area and improve the efficiency of alkaline activation, a particle size reduction process was conducted. This involved milling the material using a ceramic alumina ball mill until it achieved a small particle size to pass through a No. 325 ASTM E11 standard sieve, corresponding to particles smaller than 45 µm. The grinding process was carried out at 60Hz for 5 hours, with 500 g batches of the material.
Ordinary Portland Cement (OPC) used in this study was a general-purpose grey cement that meets the requirements of the Colombian Technical Standards NTC 121 (see **Table 1**.) [21]. As aggregate, thick

tailings (TT) larger than 45 µm, construction sand (S), and recycled polymer (P) smaller than 5 mm were used. The AAHC-MT samples were compared with a conventional mortar (OPC + construction sand + water) as a reference.

Table 1. Standard physical requirements of cement (OPC) used

General Construction Cement Type	Value
Fineness (cm^2/g)	Minimum 2.800
Autoclave length change (%)	0.8
Air content of mortar volume (%)	Maximum 12
Mortar bar expansión-14 days (%)	0.02

2.2. Mortar mix design and Compaction

2.2.1 Mortar mix design

The AAHC-MT geopolymeric mortars were manufactured using cylindrical molds, with specimens measuring 38 mm in diameter and 70 mm in height, maintaining a diameter/height ratio of 1:2, in accordance with INV 142-13 [22] (see **Figure 2**). The Si / Al and Na / Al molar ratios were adjusted by the addition of alkaline activators, specifically Na_2SiO_3 and Na_2CO_3, at concentrations of 7 M and 11 M, respectively according with the literature[23-25]. Three sample blocks were prepared: one with added S alone, another with 30% TT, and a third with 15% TT and 15% P. Additionally, a mixture of S + OPC without mine tailings was employed as a reference. The samples were cured at room temperature and evaluated in terms of mechanical strength at 7, 14 and 28 days, with each block of samples manufactured in triplicate, see the mix design in **Table 2.**

Table 2. Mix design

Sample	Fine Aggregate	Thick Aggregate	OPC	Molar Relations	
				Si/Al	Na/Al
S	10%	77%	9%	2.7	0.5
TT30		47%			
TT15-P15		47%			

2.2.2 Compaction

To prepare the AAHC-MT test specimens, it is essential to determine the optimal moisture content, which allows the soil to achieve maximum dry density and, consequently, greater strength. This process began with the INV E-141-13 test, "Moisture-Dry Unit Weight Ratio in Soils (Standard Proctor Test)." Method A from the standard was employed since over 80% of the soil passed through sieve No. 4. For the test, a 16 kg dry soil sample was divided into four samples, each with moisture contents of 5%, 10%, 15%, and 20%, respectively. Each subsample was mixed with water and left to rest to ensure uniform moisture distribution throughout the soil.

The samples were then compacted in three individual layers, each receiving 25 blows, within a mold of 101.6 mm in diameter and 116.4 mm in height, as specified by the standard. The compaction was performed using a standardized hammer weighing 14.5 N with a controlled drop height of 305 mm, resulting in an approximate compaction energy of 600 kNm/m³. The wet weight, dimensions, and moisture content of each sample were measured to calculate the dry unit weights. This data was used to plot the moisture-density curve, allowing for the identification of the maximum dry unit weight and the corresponding optimal moisture content. Cylindrical samples of AAHC-MT were made to perform unconfined compressive strength tests.

Figure 2. Alkali-Activated Hybrid Cement from Mining Tailings and OPC.

2.3. Characterization.

2.3.1 Laser Diffraction Method

The particle size distribution of the mining tailing was determined using the laser diffraction method. The device used for this was a Master Sizer 2000 E, Single Narrow model, while an aqueous solution of was used as a dispersant, with Hydro 2000 (MU)A accessory.

2.3.2 X-ray diffraction XRD

The tests were performed on a Malvern-PANalytical X-ray diffractometer (XRD) Model Empyrean 2012, with a 3D Pixel detector and a Cu source (λ=1.541 Å) at 45 kV and 40 mA; Goniometer: Omega/2 theta and platform configuration: Spinner with 4 s rotation, the step was 0.05° for standard measurements, a time per step of 50 s. The analysis was performed on the XRD patterns and compared to the references and standards found in HighScore Plus software from PANalytical version 3.0. Both the MT and the AAHC-MT were analyzed with this technique.

2.3.3 X-ray fluorescence

With Malvern-PANalytical Model Zetium mineral edition 4kW equipment, the samples were analyzed and directly measured using Uniquant software. The analysis corresponds to a quantitative measurement; the result is reported on a dry basis. This application has a detection limit of 0.1 %.

2.3.4 Scanning Electron Microscopy SEM and Field Emission Scanning Electron Microscopy FESEM

The samples were fixed on a graphite tape, covered with a thin gold coating (DENTON VACUUM Desk IV equipment). Mining tailings were examined with a high-vacuum scanning electron microscope (JEOL JSM 6490 LV) to capture high-resolution images. Due to the nature of the samples, a backscattered electron detector was used to assess their morphology. AAHC-MT samples were similarly analyzed using a high-vacuum field emission scanning electron microscope (FESEM) Thermo Fisher Scientific Scios 2 LoVac, also to obtain high-resolution images. In this case, the backscattered electron detector was used not only to evaluate morphology but also to highlight differences in chemical composition. Elemental analysis was conducted using a Microanalysis System (UltraDry 129 eV, 30 mm², model SDBX-30PM-B).

2.3.5 Mechanical Characterization by Unconfined Compressive Strength (UCS)

The Unconfined Compressive Strength tests of the AAHC-MT samples and the reference of a conventional mortar were carried out in a Shimadzu series AGX device with a load of 5 kN and a load velocity of 2.4 kN / s.

3. Results

3.1 Mining tailing characterization

The particle size distribution of MT is shown in **Figure 3**, where a homogeneous monomodal sample distribution with a size of 20 µm can be observed. However, most of the particles are even below this size with a d (0.5) of 14.958 µm, which indicates that they have the required size to be used as precursors in a geopolymer.

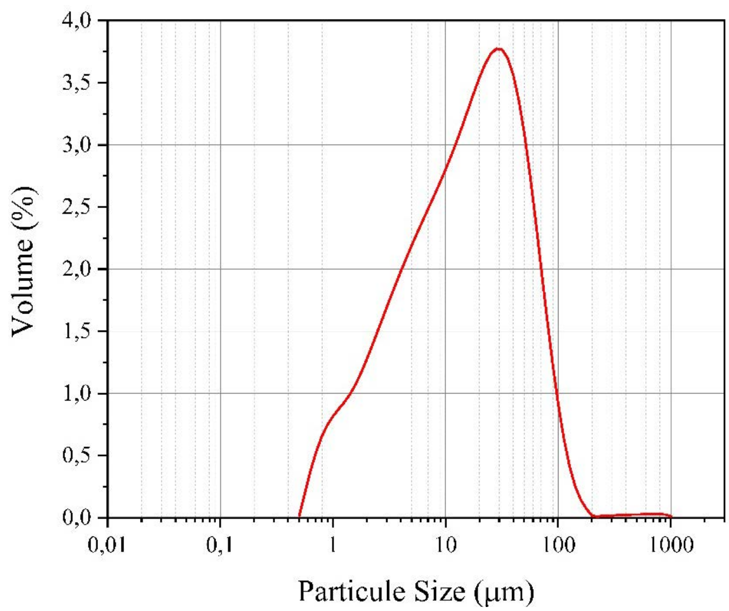

Figure 3. Particle size distribution of mining tailings by Laser Beam Diffraction (LBD).

In order to determine the presence of mainly SiO_2 and Al_2O_3 required for the generation of AACH, the MT sample was analyzed by XRF, the results presented in **Table 3** show a high percentage of SiO_2, followed by Al_2O_3 and FeO. Therefore, the MT sample chemically contains the elements required to be used as a precursor in the formation of AACH, i.e., high silicon and aluminum content.

Table 3. Chemical composition of the mining tailings sample by X-ray Fluorescence

Oxide	SiO_2	TiO_2	Al_2O_3	FeO	K_2O	Cr_2O_3	ZrO_2	Na_2O
Composition (% w/w)	68.31	0.82	22.39	2.79	2.60	0.01	0.02	0.30

The **Figure 4** illustrates the X-ray diffraction results obtained from the mine tailings. The presence of quartz (SiO_2) is clearly evident, characterised by very well-defined and high-intensity peaks indicating high crystallinity. The presence of montmorillonite ($Al_4Si_8O_{24}Ca$) and albite ($AlNO_8SiO_3$) [26-28], which contribute both silica and aluminium content, is also detected.

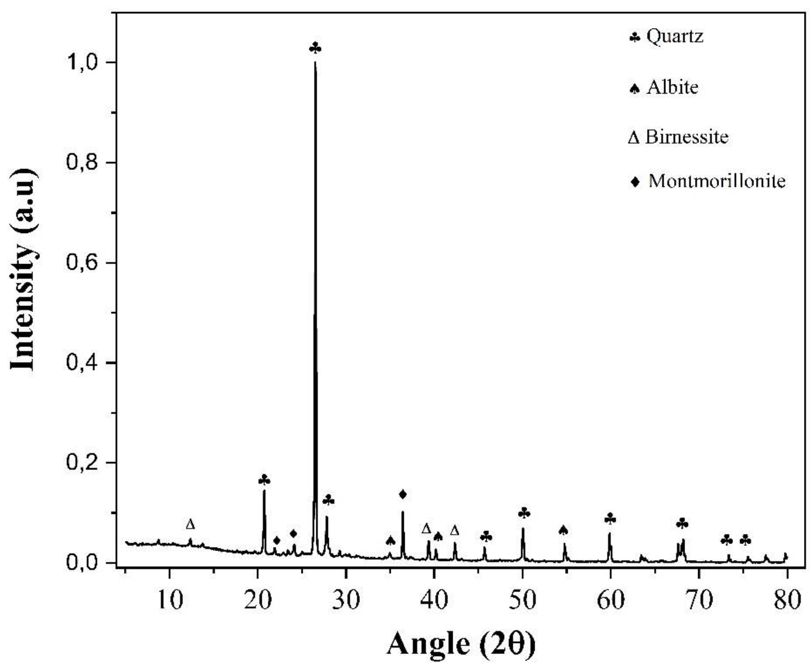

Figure 4. DRX results of mining tailings.

The morphology of the tailings is illustrated in **Figure 5**. It can be observed that the structure is highly irregular, exhibiting both pointed and rounded shapes. This can be attributed primarily to the comminution process applied. During the stages of crushing and grinding, the reduction in particle size occurs primarily due to impacts, which can result in the aforementioned structures. The particles observed in the SEM showed a wide size distribution, as can be seen in the image, with some large particles larger than 40 µm, although these do not represent the majority of the sample. This behavior is in good agreement with the results obtained by LRD

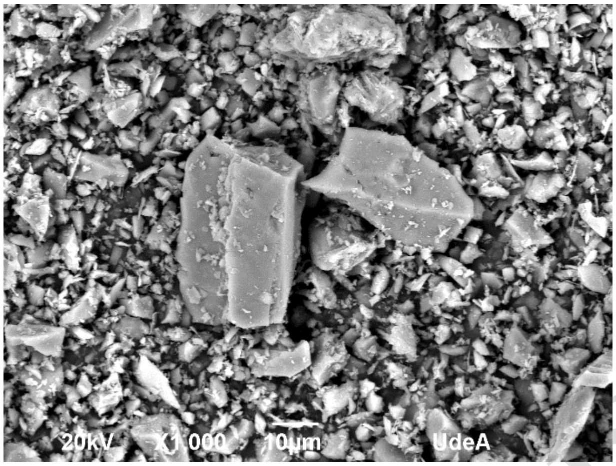

Figure 5. Surface morphology of the mine tailings of Antioquia.

3.2 Mechanical characterization of AAHC-MT

Figure 6 presents the results of the unconfined compressive strength (UCS) for the three samples evaluated (S, TT30, and TT15-P15), as well as for the reference, which corresponds to a conventional mortar, all measured at 7, 14, and 28 days of curing. The highest strengths for the AAHC-MT samples were obtained with the sand and coarse tailing aggregates, reaching 0.58 MPa at 28 days, demonstrating their potential as precursors in the production of geopolymers. In all cases, strength was increased as the curing time progressed.

However, the decrease in strength in the TT15-P15 sample compared to the other AAHC-MT samples is evident. This suggests that, although it is possible to encapsulate the recycled polymer within the geopolymer, its inclusion reduces the mechanical strength. This behavior can be attributed to the size close to 5 mm of the polymer used, which probably generates large voids in the compaction of the geopolymer.

Compared to the reference sample, which has a strength greater than 1 MPa, the AACH-MT showed significantly lower strengths. This could be due to the high crystallinity of the mining waste, which hinders the proper formation of the geopolymer matrix. Therefore, it would be necessary to apply pretreatments to the waste to reduce its crystallinity and improve its cementitious properties [29]. This could be achieved by modifying activator concentrations or including thermal or mechanical pretreatments of the tailings, as suggested by Lenis-Rodas et al. [30].

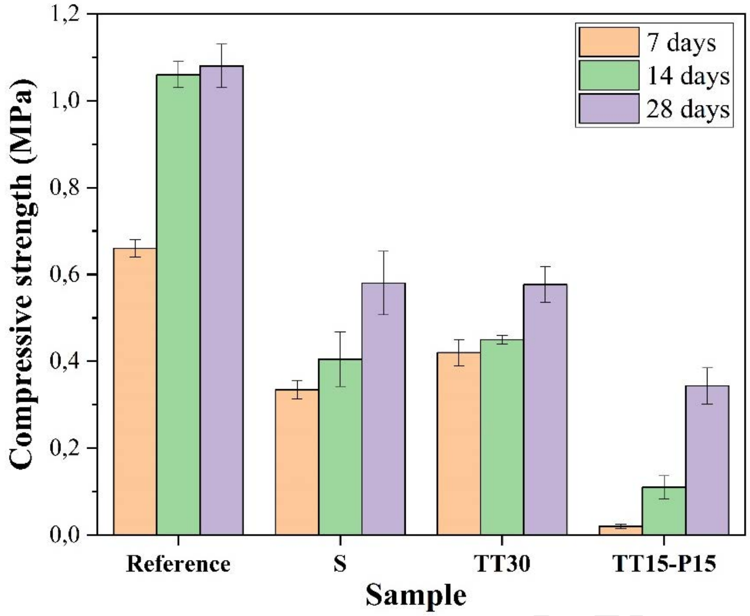

Figure 6. Unconfined Compressive Strength of AAHC-MT samples

3.3 Chemical composition of AAHC-MT

Figure 7 shows the X-ray diffraction results for the AAHC-MT samples evaluated in comparison with the reference. The phases found were Quartz (SiO_2), Montmorillonite ($Al_4Si_8O_{24}Ca$), Albite ($AlNO_8Si_3$), Calcium Alumino-Silicate Hydrate C-A-S-H, Andradite ($(Ca_3Fe^{2+})_2(SiO_4)_3$), Birnessite ($KMnO_2$), Muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$) and Sodium Alumina-Silicate Hydrate, N-A-S-H.

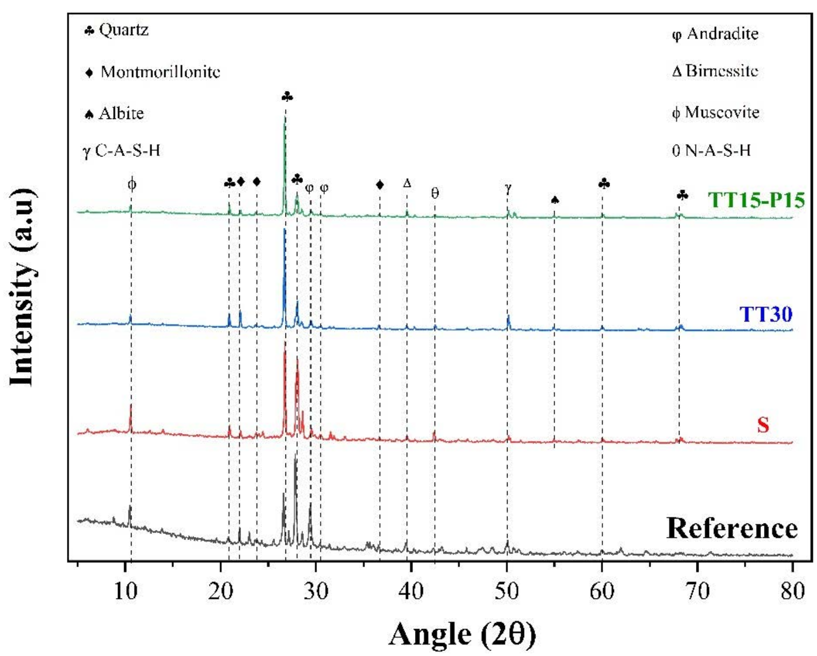

Figure 7. DRX results of AAHC-MT samples

The phases identified during the formation of the alkali activated material were N-A-S-H (sodium aluminosilicate hydrate) and C-A-S-H (calcium aluminosilicate hydrate) gels [12]. The N-A-S-H gel, the main product of the alkaline reaction in alkali activated cement systems, was observed with higher intensity in aggregate S, followed by TT30, and with much lower intensity in TT15-P15, in agreement with the UCS test results. The C-A-S-H gel, was detected in all diffractograms, covering both the AAHC-MT and reference samples. Both gels are the main products of alkaline activated cements.

Several authors have reported that the N-A-S-H gel contributes to improve the mechanical strength of alkali activated materials, since this gel fills porosity and possible cracks that may form during hardening. In addition, it has been suggested that N-A-S-H favors the formation of more ordered structures, which increases the crystallinity of the material [31]. This could explain the lower strength observed in the TT15-P15 samples compared to other mixtures, since the N-A-S-H phase was not as evident in this composition. However, few authors have reported the formation of gels for alkali activated cements with mine tailings from gold beneficiation [32]. Liu et. al report difficulty in observing C-A-S-H and N-A-S-H gels due to the amphiphilic structure and overlapping of some crystalline phases [33]. This could be similar to our case, with the peaks reported for the gels around 30°.

In addition, a change in the intensities of the main quartz (SiO_2) peaks was observed between 25° and 30°, compared to the reference, due to the different precursors used and the use of sodium silicate as one of the activators. This chemical behavior was corroborated by the XRF results, shown in **Table 4**. The increase in SiO_2 and Na_2O content in the AAHC-MT samples compared to the reference is highlighted, contrasting with the decrease in CaO, which is due to the low amount of OPC used in the AAHC-MT samples.

Table 4. Chemical composition of the mining tailings of AAHC-MT samples and reference by X-ray Fluorescence

Compo. (%w/w)	**SiO_2**	**Al_2O_3**	**Fe_3O_4**	**CaO**	**MgO**	**Na_2O**	**TiO_2**	**K_2O**	**P_2O_5**	**SO_3**
Reference	33.61	7.16	12.78	36.98	0.92	3.53	1.41	1.14	0.68	1.52
S	48.22	7.76	9.33	16.21	0.97	13.72	1.27	0.99	0.81	0.52
TT30	53.98	6.71	5.82	17.99	0.69	11.70	0.71	0.88	0.77	0.59
TT15-P15	52.36	6.18	9.29	18.95	0.77	12.98	0.96	0.94	0.80	0.54

3.4 Morphology by FESEM

Figure 8 presents the FESEM results for the AAHC-MT samples and the reference, after 28 days of curing and after failure in the compressive strength tests. In images b, c, and d corresponding to the AAHC-MT with sand, coarse tailings and recycled polymer, respectively, the presence of both the matrix and the aggregates used can be seen. It is remarkable the appearance of morphology in the form of a network or spider web, which is associated with the formation of N-A-S-H and C-A-S-H gels, products of the geopolymerization reaction. This morphology has been previously reported by other authors [34-39]

In the reference, a traditional mortar, the phases of ettringite and portlandite, characteristic of cement, were identified. However, these phases were not clearly observed in the AAHC-MT samples, possibly due to the low percentage of cement used in the mixtures. As for the EDS analysis of the AAHC-MT sample with coarse tailings as aggregate, **Figure 9**, it is observed that the areas where the network morphology is present, mentioned above, are composed of calcium, sodium, aluminum and silicon, characteristic elements of the N-A-S-H and C-A-S-H phases.

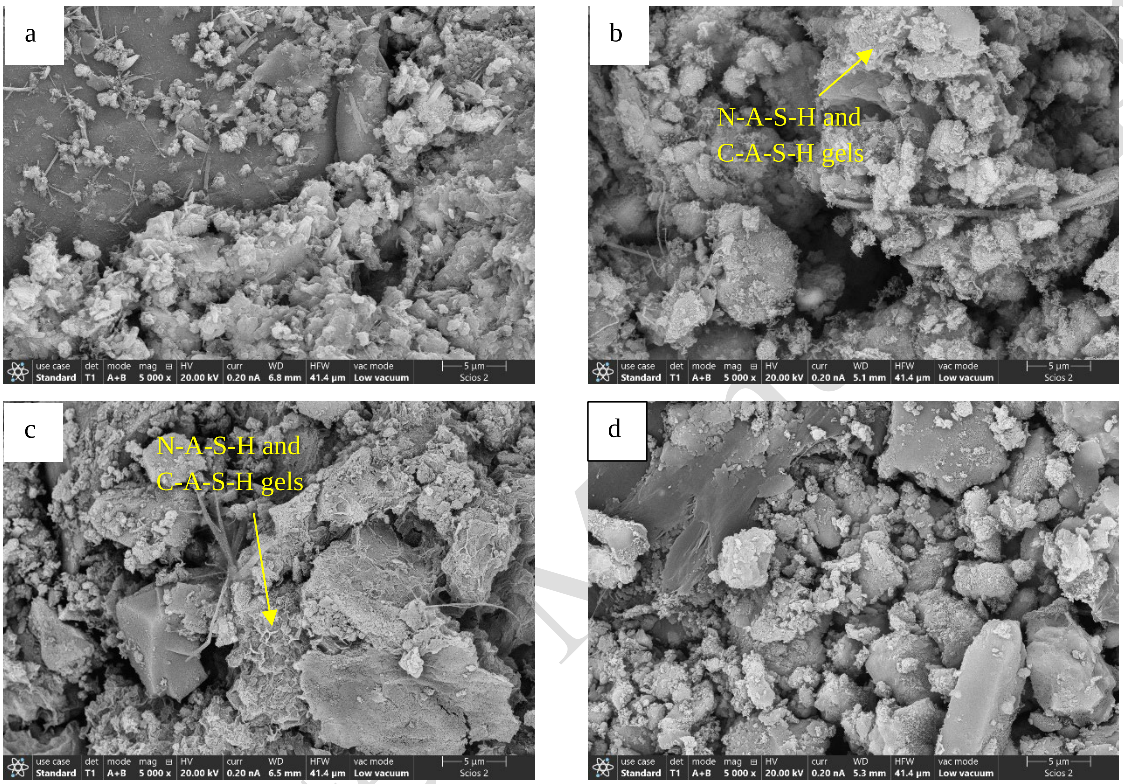

Figure 8. Morphological analysis by FE-SEM of AAHC-MT samples: **a.** Reference, **b.** S, **c.** TT30 and **d.** TT15-P15.

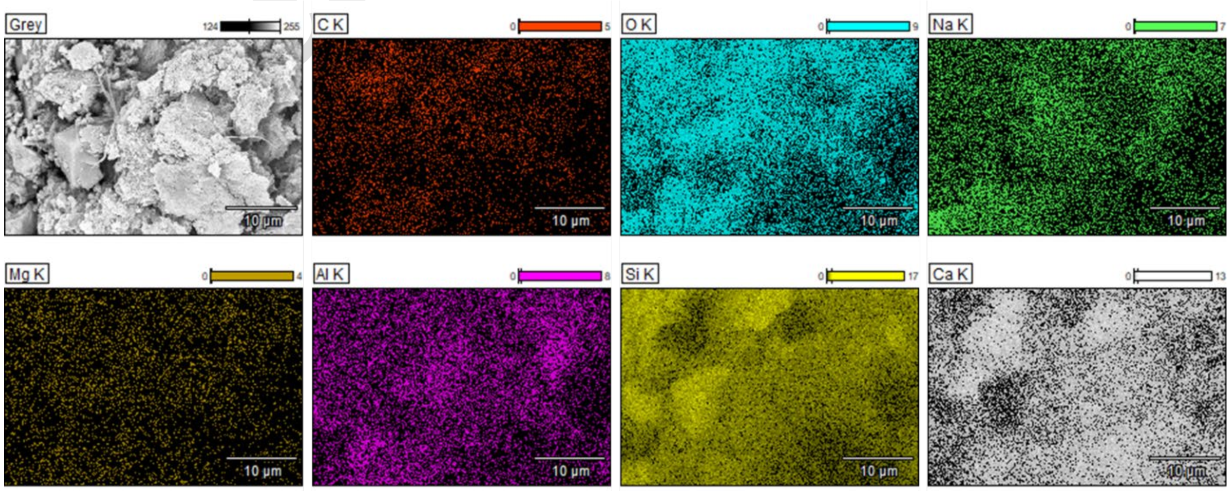

Figure 9. Analysis by EDS AAHC-MT with TT30 as aggregate.

4. Conclusions

The tailings evaluated from a mining area of Antioquia-Colombia, possess the chemical characteristics required to be used as precursors in the manufacture of AACH, i.e., they have high silica and alumina content, components necessary for alkaline activation. As expected, mining tailings are highly crystalline materials, which makes alkaline activation difficult. In the hybrid cements, strengths of up to 0.58 MPa were obtained after 28 days of curing, showing their viability for several applications in construction. By means of XRD, the formation of CASH and NASH gels characteristic of alkaline activation, were evidenced. Additionally, by means of FE-SEM it was possible to confirm the morphology of these gels.

The results of the mechanical properties of the AAHC-MT obtained, although not comparable to the reference, a conventional mortar, are promising. It is known that mine tailings are highly crystalline so these properties can be improved by further research, i.e., evaluating other factors such as activators and their molarity, further tailings size reduction for better alkaline activation, other alkaline activators or even thermal pretreatment of the tailings. Likewise, the use of OPC or other cementitious in higher proportions can be evaluated.

This work seeks to contribute to the state of the art of alkali activated hybrid cements from gold mining tailings, taking into account the few reports found on the subject. Likewise, it is desired to continue with the research to obtain a material that can be used in construction for the improvement of housing in mining areas.

This work aims to contribute to the state of the art on hybrid cements activated with alkali from gold mining tailings, considering the scarcity of studies on the subject. It also seeks to open new opportunities for the valorization of mining waste, thus promoting the circular economy in the construction sector. In addition, it is intended to continue the research in order to develop a material that can be used in the improvement of housing in mining areas of Antioquia, Colombia.

Declaration of competing interest

We declare that we have no significant competing interests including financial or non-financial, professional, or personal interests interfering with the full and objective presentation of the work described in this manuscript.

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Author contributions

M. Isaza-Ruiz, M. Restrepo-Posada, M. A. Gómez-Botero: Conceived and designed the analysis. M. Isaza-Ruiz and M. Restrepo-Posada: Collected the data. M. Isaza-Ruiz, M. Restrepo-Posada, M. A. Gómez-Botero and H. Cardona-Trujillo: Contributed data or analysis tools. M. Isaza-Ruiz and M. Restrepo-Posada: Performed the analysis. M. Isaza-Ruiz, M. Restrepo-Posada, M. A. Gómez-Botero and H. Cardona-Trujillo: Wrote the paper

Data availability statement

where the data associated with a paper is available, and under what conditions the data can be accessed. They also include links (where applicable) to the data set

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