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The Effect of Additional Pineapple (*Ananas comosus* L. Merr.) Peel Pectin on the Characteristics of Coal Fly Ash Geopolymer

Nuni Widiarti[1*], Nandia Salsa Restu Mulyani[1], Ella Kusumastuti[1], Ridho Prasetyo[1,2]

[1]Chemistry Study Program, Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Negeri Semarang,Semarang, Indonesia
[2] Chemical Engineering Study Program, Department of Chemical Engineering, Faculty of Engineering, Universitas Diponegoro,Semarang, Indonesia

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ABSTRACT

Fly ash-based geopolymer concrete is a candidate for a more sustainable material than concrete, with lower embodied energy and high early-age compressive strength properties. This work has focused on studying the use of pineapple peel pectin as an additive to enhance the compressive and split tensile strength achievable in a geopolymerization process. The compressive strength and split tensile strength were tested by physical testing, and the mineral phases, functional groups, and microstructure were analyzed by chemical analysis (XRD, FTIR, and SEM-EDX). Geopolymers containing 0%, 1%, and 2.5% pectin were fabricated. The surprising optimum was the 1% variation, which reached compressive strength of 22.13 MPa and split tensile strength of 3.18 MPa when making medium-quality concrete. XRD results of the best performing 1% sample exhibited mainly an amorphous phase, where amorphization is evident at 20–40°2θ due to broad signal peaks, a sign of successful geolypolimerization. Geopolymerization was also confirmed by FTIR analysis through the presence of Si–O–Si and Si–O–Al asymmetric stretching vibration peaks at 1018.41 cm^{-1}, illustrating the inclusion of pectin observed by the C–H stretches in the range 2900–2300 cm^{-1} and the carboxyl group stretch at 1635.64 cm^{-1}. The microstructure of the quality concrete 1% formulation was characterized by a tight, perfect structure as seen through SEM images, while the 0% and 2.5% mixes had more porous and cracked structures. These results suggest that 1% pectin incorporation improves geopolymer mechanical performance without adversely affecting structure, merits for further mechanical properties, and long-term stability.

**Corresponding Author: nuni_kimia@mail.unnes.ac.id*
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INTRODUCTION

The problem of carbon dioxide emission from burning limestone and clay at high temperatures in traditional Portland cement production cannot be overemphasized and needs to be focused on in environmental protection issues [1]–[3]. Various approaches have been developed to reduce CO_2 emissions of cement manufacturing, including using supplementary *cementitious materials* such as fly ash and ground granulated blast furnace slag, and *carbon capture* [4]–[6]. But these solutions have limitations, for instance,

partial cement replacement and therefore not achieving full CO_2-free end products, let alone the high costs associated with *carbon capture* technologies. Therefore, it is necessary to produce more environmentally friendly *cementitious materials*. One of the raw materials used to produce environmentally friendly concrete is *geopolymer* because it does not require cement [7]–[9].

A few studies showed the possibility of using it for high-performance building materials, *fire retardation coating*, and wastewater treatment [10]–[12]. Nevertheless, some obstacles lie in the way of further development of the geopolymer technology: variations in the composition of raw materials, long-term resistance, and optimization in large application dimensions [13]–[15]. However, these limitations must be addressed to achieve another growth phase concerning the usage area of *geopolymer*-based products. One of them, which has been subjected to significant research, is to use *geopolymers,* which do not require the limestone burning step, significantly reducing the impact on the environment [8], [16]. *Geopolymer* is seen to require far less energy production and produce significantly lower CO_2 emissions compared to conventional Portland cement-derived concrete, making it an environmentally benign alternative [17], [18]. So there are specific advantages for environmentally-friendly options, such as the *alkali-activated* and *carbon-cured* (and let's not forget *geopolymers*). However, they still require further studies on their robustness and applicability on a large scale.

Work undertaken in previous studies has indicated the durability of the *geopolymer* after immersion in 10% magnesium sulfate for 48 weeks, with *compressive strength* and *tensile strength* decreasing by 33% and 35%, respectively. *Geopolymer* is eco-friendly, employing industrial waste and by-products as raw materials, with lower energy consumption during synthesis, while developing high *compressive strength* and hardness within a few hours after casting [19]–[21]. Coal fly ash, as defined by SNI 03-6414-2002, is a finely grained output of furnaces in steam power plants (PLTU) from coal combustion, characterized by a round, smooth, and pozzolanic nature. Substituting coal fly ash in *geopolymer* synthesis improves strength and durability, exhibiting pozzolanic and *cementitious* reactions [22]–[24].

However, variations in the source and combustion method of fly ash result in *geopolymers* with differing properties. Differences in particle distribution and reactivity cause workability problems that should be considered to optimize mix proportions [25]–[27]. Similar challenges apply to long-term durability considerations, such as moisture resistance, freeze–thaw cycles, and chemical attack. Addressing these issues is critical to unlocking the potential of coal fly ash-based *geopolymer* materials for broader applications without compromising system reliability and scalability.

Coal fly ash primarily contains SiO_2, Al_2O_3, Fe_2O_3, and trace elements (e.g., carbon), along with calcium, magnesium, and sulfur from power plants [28]–[30]. An example

is the steam power plant (PLTU) Tanjung Jati Jepara in Indonesia, where the waste material achieves high *compressive strength* when *geopolymers* are synthesized from class F coal fly ash under high-temperature curing [31]. A *compressive strength* of 19.42 MPa has been achieved using *geopolymer*-based lightweight concrete made from coal fly ash cenospheres [32].

Further investigations have demonstrated improvements in mechanical properties by incorporating additives such as water hyacinth fiber and aluminum powder into fly ash-based *geopolymers* [33]. For example, varying the percentage (w/w) of water hyacinth fiber pretreated with alkaline hydrolysis resulted in *compressive strength* and *split tensile strength* values of 27.57 MPa and 24.11 MPa, respectively [33]. In addition to fly ash as a prime precursor, alkali *activators* such as sodium silicate and sodium hydroxide are necessary binders. Incorporation of cellulose polysaccharides has also been shown to enhance mechanical properties, particularly *compressive*, *tensile*, and *split tensile strengths* [34]–[36].

Cellulose fibres are also incorporated to improve interfacial adhesion between fibre and matrix, promoting the mechanical performance of the polymer by resisting the further propagation of cracks [38]–[40]. However, the use of cellulose as reinforcement is limited by non-uniform fibre distribution [39], [40], degradation of fibres under harsh environmental conditions [40], and inconsistent performance of cellulose due to varying cellulose morphology and chemical composition [60], [61]. To avoid these drawbacks or improve microstructure properties of the *geopolymer*, alternative polysaccharide sources with low cost and wide availability were sought, including pectin extracted from pineapple peel [16].

Introducing 0.2% (w/w) pineapple leaf fibre into coal fly ash-based *geopolymer* gave a *compressive strength* of 41.91 MPa and *split tensile strength* of 16.28 MPa [42]. Its *compressive* and *split tensile strengths* with a content of 0.2 g of water hyacinth fibre and aluminium powder were 32.03 MPa and 22.91 MPa, respectively [33]. Similarly, the mechanical capabilities were also improved by the use of banana fibrous stem, where the *compressive strength* of BFS is 32.35 MPa and *split tensile strength* is 10.9%, compared to fiber mixtures, and homogeneity was found in fiberless samples [43].

Some polysaccharides (that is, pectin) are denoted [44], [45] to enhance the *compressive* and *tensile strength of geopolymers*. Even though pineapple contains up to 29% pectin [46], pineapple peels are a potential sustainable source of pectin in addition to utilization in fertilizer [46]–[50], animal feed [50], [51], and bio-ethanol production [52], [53]. Pineapple peel fibres are found to have a *tensile strength* up to 1058.06 MPa, are also high in cellulose content, and thus are suitable for use as a reinforcement to enhance the strength of the *geopolymer* matrix, paint layers, as well as being biodegradable and inexpensive [39], [57], [59].

The gelling power of pectin is believed to increase the *viscosity* of the *geopolymer*, and therefore, is expected to influence the *setting time*, adherence, and microstructural density [54], [55], [63]–[65].

While some natural fibres like banana and hyacinth have been indicated, few studies on the direct use of pineapple peel pectin in polymer composites have been reported. No study to date has sorted these parameters, and the present study tries to sort the mechanical and structural effects of pectin incorporation, with the hope that the findings may open up an avenue to explore new uses in bio-based materials for optimizing *geopolymer* performance in the future [61], [62].

METHODS

1. Materials and Tools

The orthogonal Portland cement (POC) itself is a type 1-ordinary Portland cement, meanwhile, the aggregates are the ones from Serayu River; the *alkali activator* used are Na_2SiO_3 and NaOH (96.7%); the sand used has a fine-grained class as fine aggregate also the coarse aggregate used in this study is gravel with a nominal maximum size of 20 mm, the chemicals and tools such as: Na_2SiO_3·pentahydrate, NaOH, H_2O, HCl 1 N, 96% technical ethanol and glass wares (beaker glass, stirring rod) and the NaOH used in the POC testing tube (Pineapple from Land piece) from Pemalang.

2. Equipment

The instruments listed below are used in the synthesis of the *geopolymers* – they are 100 and 60 mesh (Tatonas) dater for (using), analytical balance, beaker in glass (Pyrex), the blender, the thermometer, and the chronometer, user, user the plastic container, the stirrer (mixer), the plastic mold of cylinder (for the best control the *geopolymers* to mould), (as a container that has the mold of in the oven) onto and the oven (removes the content of water). The equipment Characterization of *Geopolymers compressive strength* testing machine and *tensile strength* testing machines Universal Testing Machine Shimadzu AG-X, X-ray Fluorescence (XRF) Bruker S1 Titan, X-Ray Diffraction (XRD) Bruker 6000 diffraction angle 2θ (10°–60°), Fourier Transform Infrared (FT-IR) IR Prestige-21 Shimadzu wavelength 400–4000 cm^{-1}, Scanning Electron Microscopy – Energy Dispersive X-ray Spectroscopy (SEM-EDX) Detector – JEOL JSM-6360LA.

3. Material Preparation: Pectin from Pineapple Rind

A 100-gram sample of the pineapple skin is weighed, homogenized with dried pineapple skin to a smooth paste, and screened using a 60-mesh sieve. Extraction: The finely sifted pineapple skin was loaded onto a three-neck flask,1 and 1 liter of 6 M HCl was added to the flask [66], [67]. The heating was activated with an electric heater while stirring was carried out using a magnetic stirrer at the rate of 300 rpm at 90°C for 3 hours, using a water bath [66], [67]. The pectin extract was concentrated by heat treatment (90°C) and vigorous stirring to half of its original volume, and the pectin was recovered from the filtrate [68], [69]. The resulting filtrate is cooled to room temperature, after which it undergoes precipitation by adding pectin filtrate with a volume ratio of 1:1, then stirring well and allowing to stand for 12 h until pectin crystals form. Thereafter, pectin is dried at 40–50°C for eight h [70].

4. Materials and Tools

The Study materials Type C Coal Fly Ash: The Silica in this study was extracted from a steam power plant (PLTU), Tanjung Jati B, Jepara. *Alkali activator*: Sodium silicate (Na_2SiO_3) technical, Sodium hydroxide (NaOH 96.7%) technical, Distilled Water (H_2O), technical, 96% ethanol. Pineapple skin: Pineapple skin was collected from Pemalang.

5. Equipment

Several equipment that should be used to synthesize *geopolymer* were: 100 and 60 mesh (Tatonas) for standardizing, analytical balance, glass beaker (Pyrex), blender, thermometer, stopwatch meter, plastic stirrer, plastic container, stirrer (mixer), plastic cylinder mould (to mould a *geopolymer*), baking sheet (a container when in oven), and oven (removes water). *Geopolymer* characterizations such as *compressive* and *tensile strengths* can be performed using various tools such as Universal Testing Machine Shimadzu AG-X range, X-ray Fluorescence (*XRF*) Bruker S1 Titan, X-ray Diffraction (*XRD*) Bruker 6000 (diffracted angle 2θ 10°–60°), Fourier Transform Infrared (*FT-IR*) IR Prestige-21 Shimadzu (wavelength 400–4000 cm^{-1}), and Scanning Electron Microscopy – Energy Dispersive X-ray Spectroscopy (*SEM-EDX*) Detector JEOL JSM-6360LA.

6. Preparation of Pineapple Peel Pectin Comprising Its Functional Groups

Dried pineapple skin slurry is filtered through a 60 mesh strainer, and 100 g of sample (pineapple skin) is weighed. The extraction was conducted by 1 L of 6 M HCl of acid-reinforced pineapple peel in a three-neck flask [66], [67]. A water bath with electrical heating equipped with stirring by magnetic stirrer at 300 rpm and 90°C for 3 hours was performed [66], [67]. The pectin filtrate was concentrated by heating on a water bath at 90°C with vigorous stirring until it was reduced to half the initial volume [68], [69]. The filtrate was cooled to room temperature; precipitation was performed by adding alcohol to the pectin filtrate (volume ratio of 1:1), stirred well, and allowed to stand for 12 h to produce pectin. The obtained pectin was dried at 40–50°C for 8 hours [70].

7. Coal Fly Ash Preparation

Type C coal fly ash was collected from the steam power plant (PLTU) Tanjung Jati B Jepara. Thereafter, the fly ash was sieved using a 100 mesh sieve and oven-dried at 105°C for 24 hours to remove its air content. The *XRF* technique [71] was employed to study the fly ash for elemental analysis.

8. Alkali Activator Preparation

The test sample prepared with glassy water was allowed to stand separately for at least 24 h. Because the dissolution process of NaOH is exothermic, NaOH was dissolved in distilled water first. Then, at least 24 h was needed for the temperature of the NaOH 8 M solution to stabilize [72]. To standardize the NaOH solution state (reaction with CO_2 in the air), it also underwent curing for 24 h. After 24 h of treatment, an amount of Na_2SiO_3 was added, and the same treatment was applied as that for the NaOH solution [71], [73]–[75].

9. Preparation of the Geopolymer with Pineapple Peel Pectin

The *geopolymer* was derived from coal fly ash, pineapple peel pectin, and *alkali activator* solution. The blend is washed and homogeneously mixed and kneaded. It was then poured into a cylindrical mould with a ratio between height and diameter 2:1. Mold removal from the test specimen was performed after 1–3 days. After demolding, the samples were placed in a casserole and wrapped to protect them from exposure using plastic film. Samples still covered with plastic were dried in an oven at 60°C for 24 h and then stored for 28 days [22], [76].

Table 1. Geopolymer Variations with Variations in Pineapple Peel Pectin Addition

No	Coal fly ash (g)	NaOH (g)	H2O (g)	Na_2SiO_3 (g)	Pineapple peel pectin compared to fly ash (% w/w)	Weight of pineapple peel pectin (g)
1.	33	4	8	10	0,00	0,00
2.	33	4	8	10	0,50	0,165
3.	33	4	8	10	1,0	0,33
4.	33	4	8	10	1,5	0,495
5.	33	4	8	10	2,0	0,66
6.	33	4	8	10	2,5	0,825
7.	33	4	8	10	3,0	0,99

10. Compressive Strength Tests

Geopolymer compressive strength test: The *geopolymer compressive strength* was conducted using a *compressive strength* testing machine (Universal Testing Machine) at the Structure Laboratory of the Civil Engineering Department, UNNES. All of the diameter and height of the tested cylindrical geopolymers (1:2). The compressive strength test was determined using the 28-day aged *geopolymer* test specimens—*compressive strength* calculated according to ASTM C39M.

11. Split Tensile Strength Test

The Torsee Universal Testing Machine measured the split tensile strength of geopolymers at the Structure Laboratory of the Civil Engineering Department, Universitas Negeri Semarang. ASTM C496/C496M — Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete ACI TERM Volume 118 Issue 3 MC Members 50PIC Chapter 10 — Agreement about the Relationship between *Tensile Strength* of Cylindrical Concrete Members 67 Specimens. The samples were placed flat when evaluated. Vertical load was applied gradually in the form of a cylindrical load. Maximum mark and splitting occurred due to horizontal pull loading at 0.7–1.4 MPa/min. The test was repeated three times, taking the average. Comparison of the 28-day-old *geopolymer* specimens was made based on the *split tensile strength*. *Tensile strength* was tested according to ASTM C469M.

12. Characterization

FTIR Results: FTIR determined the properties and functional groups of the synthetic geopolymer. Up to 2–3 mg of sample per 200–300 mg KBr was used to prepare a *geopolymer* specimen using the KBr Pellet method. Results include absorption intensity (T%) and wave number (cm^1). OH or water groups in the *geopolymer*

were obtained from the *FTIR* wavelength range of 3500–3200 cm^{-1} to 1600 cm^{-1}.

The lowest *tensile strength* after 28 days of age of fly ash *geopolymer* samples tested was observed in this test [43]. Maximum *compressive strength* was reached at the end of 28 days, while the mineral phase of the *geopolymer* was identified by *XRD* (X-ray Diffraction) analysis. The laboratory used was the Institute of Technology Sepuluh Nopember (ITS). For morphological tests of the samples with the highest pectin concentration from pineapple peel showing the best *compressive strength* after 28 days of storage, *SEM* (Scanning Electron Microscopy) was used with magnifications of 2000×, 5000×, and up to a maximum of 10,000×. All samples were in flake form, and tests were conducted at the Laboratory of the Institute of Technology Sepuluh Nopember.

RESULTS AND DISCUSSION

1. Synthesizing the *Geopolymer*

The Type F coal fly ashes used in this work are from the steam power plant (PLTU) Tanjung Jati B, Jepara. The fly ash as supplied was wet, blackish grey, and moist by nature, and it partly contained some coarse aggregates similar to gravels (Figure 1). Type F fly ashes (CaO < 20%) were used, one of which is self-cementing and more reactive [77]. It has been reported that Type C fly ash with high calcium content can accelerate geopolymers' setting time and early-age compressive strength [78], [79]. However, it produces a far more stable *geopolymer*-type F fly ash with a high amorphous percentage [80], [81].

Although coal fly ash has a range of inhomogeneous characteristics, XRF must characterize it to analyze its chemical constituents. The samples of the coal fly ash are darker in color, brown, smaller in size, 149 microns, and with smooth morphology. XRF characterized the mineral composition of coal fly ash (Table 2).

Table 2. Results XRF Analysis

Identified compounds	XRF Analysis (%)
SiO_2	30.4
Al_2O_3	13.7
Fe_2O_3	11.3

Then, based on the CaO content contained in coal fly ash from Tanjung Jati B Jepara coal, it belongs to the group of coal fly ash class F because of the content of <10% CaO (ASTM C-618-03, 2003). The activating solution *alkali* content is prepared with sodium hydroxide (NaOH) [72], distilled water (dissolvent) as well as technical sodium silicate (Na_2SiO_3) [71], [73]–[75]. Si and Al minerals of coal fly ash are leached by NaOH to form silicate and aluminate monomer ions, which contribute to the formation of the *geopolymers* [9]. Sodium silicate (Na_2SiO_3) serves to increase the level of soluble Si for the provision of a sufficient amount of silicate oligomers for the growth of the chain. It may enhance the compressive strength value and densify the *geopolymer* paste [82], [83].

Previous research explains the *compressive strength* properties of *geopolymers with coal fly ash and alkali activators, which consist* of sodium hydroxide and sodium silicate. The optimum ratio was found to be 3:1 when NaOH was 8 M, the water factor was 0.2, and the maximum *compressive strength* obtained was 39.147

MPa [84]. *Geopolymer* (82% coal fly ash/ 16% *alkaline activator* solution with pineapple peel pectin (2%)).

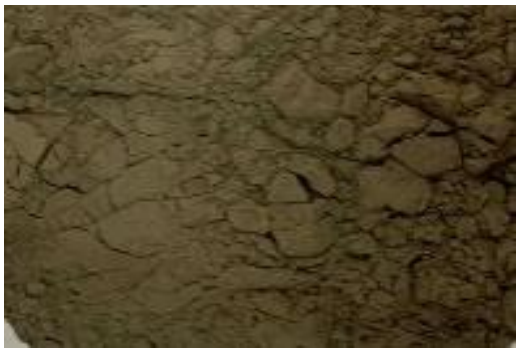


Figure 1. Appearance of Fly Ash

Figure 2. Results of geopolymer synthesis with the addition of pineapple peel pectin variations (a) 0%; (b) 0.5%; (c) 1%; (d) 1.5%; (e) 2%; (f) 2.5%; (g) 3%.

Geopolymer used pineapple peel pectin in a 0–3% mass variation. Nonetheless, the composition of pectin from pineapple peel added at different ratios to get the optimum composition that could improve the mechanical properties and durability of *geopolymer* to some extent should be clearer [61], [62], [85]. It is noted that the decay of a particular material may be a function of the concentration applied [81]. Still, pectin may have various effects at various concentrations. The threshold is important because pectin can no longer provide any strength after reaching this value, and the deterioration becomes marked when the organic matter content is too high [86]. Furthermore, this modification unveils the role of pectin with the *geopolymer* matrix and its effect on the material's distribution, adhesion, and physical and chemical characteristics [87], [88].

The *geopolymer* containing pineapple peel pectin was varied in mass (traditional 0–3%) as shown in Fig. 2. These differences were introduced to determine the best pineapple peel pectin incorporation in *geopolymer* based on physical integration of *compressive* and *split tensile strength* properties. It still contains pores created by the air bubbles in the *geopolymer*. It is the same color throughout the *geopolymer*. The pure white color is impossible since the sodium silicate has not been mixed uniformly in 0% *geopolymer* variation: 0.5% and 2.5%. In the case of 0.5 %pectin variation, the principal factor for *geopolymer* cracking is excessive evaporation during oven curing. Other studies have also demonstrated moisture loss due to increased temperature, leading to shrinkage-related cracking in *geopolymer* systems with some organic additives [89]–[91]. There might also be a high binder interaction of pectin with the *geopolymer* matrix. An excess amount of this organic content can affect *polymerisation* discontinuity, causing a reduction in bonding of the aluminosilicate chain.

Geopolymer systems with 1%, 1.5%, 2%, and 3% pectin may exhibit smoother structure and surfaces at the micro level of pores (as follows in the SEM Fig. 5 analysis). The pores and hence flake sizes are easily evident in the 0% and 2.5% cases (Fig. 2). Even if the pectin gel locally runs on the surface of the geopolymer, a consistent and

homogeneous distribution throughout the geopolymer matrix is an undetectable phase.

Geopolymers were fabricated from pineapple peel pectin in a 0–3% mass ratio variation. However, the composition of pectin extracted from pineapple pulps was varied and needs to be clarified to identify the suitable composition to improve the mechanical properties and durability of *geopolymers* to some extent [61], [62], [85]. Due to various concentrations, a threshold level must exist above which pectin is ineffective, and too much organic matter decreases strength [86]. Furthermore, this difference provides insights into the interaction of pectin with the *geopolymer* matrix, the effect on distribution, adhesion, and the physical and chemical properties of the material [87], [88].

The *geopolymer* containing pineapple peel pectin was varied in mass (range 0–3%) as presented in Figure 2. These differences were used to seek the best pineapple peel pectin geopolymer based on PIFS performance (referring to the physical merge of *compressive strength* (CS) and *split tensile strength* (STS)). The air bubbles that form the pores are still present in the *geopolymer*. The color of the *geopolymer* is uniform and consistent throughout. A perfectly white color remains impossible because sodium silicate had not blended well under the 0% *geopolymer* variation: 0.5% and 2.5%. In the case of 0.5% pectin variation, the cracking of fly ash-based *geopolymer* occurs mainly due to overdrying during oven-curing. It has also been demonstrated by other related studies that an increase in temperature could lead to fast moisture loss and shrinkage-related cracking in *geopolymer* systems incorporated with certain organic additives [89]–[91]. It is also possible that pectin might interact with the *geopolymer* matrix at high binder filling. This extra organic material can interfere with *polymerisation* and has the potential to act as a barrier for aluminosilicate chain formation.

Geopolymer systems with 1%, 1.5%, 2%, and 3% pectin may exhibit smoother structures and surfaces on the microscale of pores (as shown in the SEM analysis, Figure 5). The pores and flake sizes are more evident in the 0% and 2.5% cases (Figure 2). Even if the pectin gel locally runs on the surface of the *geopolymer*, a consistent and homogeneous distribution throughout the matrix is undetectable.

2. FTIR Analysis

After synthesizing the *geopolymer* with pineapple peel pectin from 0% to 2.5%, the FTIR spectrum was used to investigate the functional groups in the resulting *geopolymer*, as illustrated in Figure 3. The peak width is located at the wavenumber 3425.58 cm^{-1} in the absence (0% variation) of pectin from pineapple peel, followed by small peaks at 2924.09 cm^{-1}, 2854.65 cm^{-1}, and 2376.3 cm^{-1}, which are markers of *stretching* vibrations (–OH, H_2O).

The addition of 1% pineapple peel pectin in the *geopolymer* causes a broad peak at 3425.58 cm^{-1}, with broader peaks appearing at 2939.52 cm^{-1}, 2376.3 cm^{-1}, and 2345.44 cm^{-1}, corresponding to the *stretching* of –OH and H_2O [18]. In this vibrational region, the H_2O *stretching* vibration becomes narrower and the peak shifts to lower wavenumbers.

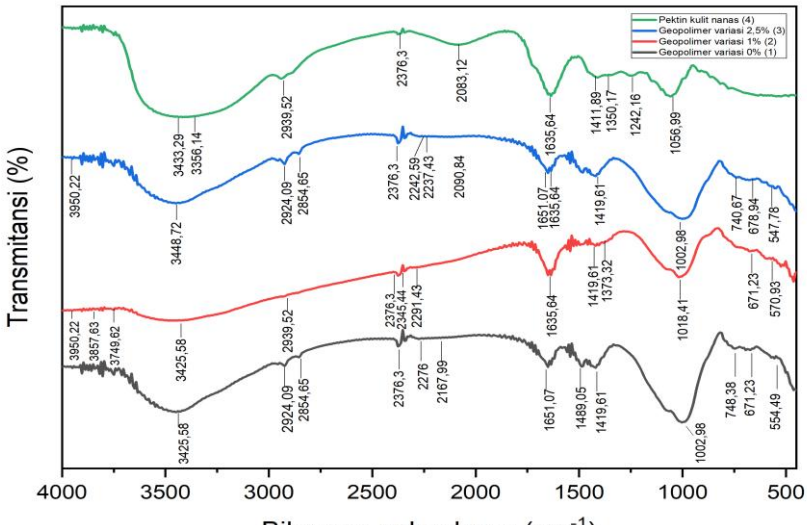


Figure 3. FTIR spectrum of geopc.y.ne. with added pineapple peel pectin (1). Variation 0%; (2). Variation 1%; (3). Variation 2.5%; (4). Pineapple peel pectin.

For the 2.5% *geopolymer* variation, the addition of pineapple peel pectin results in broadening of the peaks with a shift towards higher wavenumber at 3448.72 cm^{-1}, with other smaller peaks at 2924.09 cm^{-1}, 2854.65 cm^{-1}, and 2376.3 cm^{-1}. An absorption at wavenumber 1002.98 cm^{-1} is observed for the 0% and 2.5% variations, while for the 1% variation it appears at 1018.41 cm^{-1}. The infrared spectrum of the OCO group shows absorptions in this region, and according to Na_2CO_3 [92], the bands at 1395.17 cm^{1} correspond to the *stretching* vibration of the OCO group compared to OH and isomeric species.

The FTIR spectrum of the pineapple peel is given in Figure 3, showing absorption bands at 3433.29 cm^{-1} and 3356.14 cm^{-1}, reflecting OH *stretching* vibrations linked to hydrogen bonds, indicating an increase in hydrogen content within the *geopolymer* [4]. Monitoring by water vapor adsorption methods indicates the presence of hydroxyl functional groups on droplet surfaces even at early stages of droplet *polymerisation*, which can retain water at the droplet/drop-phase interface and improve workability [7]. Due to the hydroxyl functional groups, a setting delay may also occur.

On the other hand, excessive hydrogen bonding may interrupt gel structure development and compromise the long-term stability of the *geopolymer* [10]. Other studies have reported that increased hydroxyl content may lead to higher porosity [93] or slower condensation reactions, potentially decreasing *compressive strength* and durability [94]. The absorption band at 1350.17 cm^{-1} is attributed to the CH *stretching* vibration, and a visible shift in wavenumber is noticeable [92].

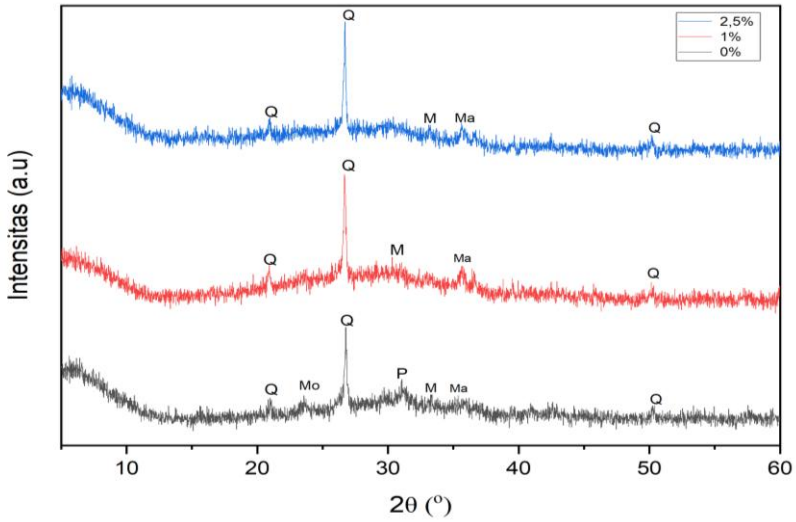


Figure 4. Geopolymer diffractogram with 0% variation; 1%; 2.5% with the addition of pineapple peel pectin, (Q) *quartz* (SiO_2), (Ma) *magnetite* (Fe_3O_4), (M) *mullite* ($3Al_2O_3.2SiO_2$), (P) *potassium* (K), (Mo) *molybdite* (MoO_3).

3. XRD and SEM EDX Analysis

The treatment in this study was conducted on the highest, intermediate, and lowest compressive strengths of a geopolymer specimen (variation of the percent addition of pineapple peel pectin: 1%, 2.5%, and 0% (without addition of pineapple peel pectin)). The image of the diffractogram pattern can predict amorphous phases in *geopolymers* based on the shape of the hump, characterized by a broad and almost irregular image, within the range of 2θ = 20°–40° [43]. Figure 4 shows the diffraction pattern on *geopolymer* molding with variation of pineapple peel pectin addition by composition of *compressive strength* for 1%, 2.5% variations, and 0% variation without pineapple peel pectin.

Concerning the *compressive strength*, peaks shifted to a higher value, and the quartz mineral (SiO_2) played a role in the 1% composition of the pectin (Figure 4) at 2θ = 20.90°, 26.68°, and 50.20° (JCPDS 96-901-0145). The mineral phase of the *geopolymer* with 1% variation by adding pineapple peel pectin shows a larger and clearer amorphous phase than the *geopolymer* without the addition of pineapple peel pectin (0% variation) and the *geopolymer* with 2.5% variation.

The addition of pineapple peel pectin resulted in the highest intensity at 2.5% variation of *geopolymer*; however, this had a negative impact, where too high intensity can lead to a decrease in the *compressive strength* and *split tensile strength* of the *geopolymer* [54], [95], [96]. Results also revealed that the 0% variation without pectin yielded a greater crystalline phase than the 1% and 2.5% variations with pectin.

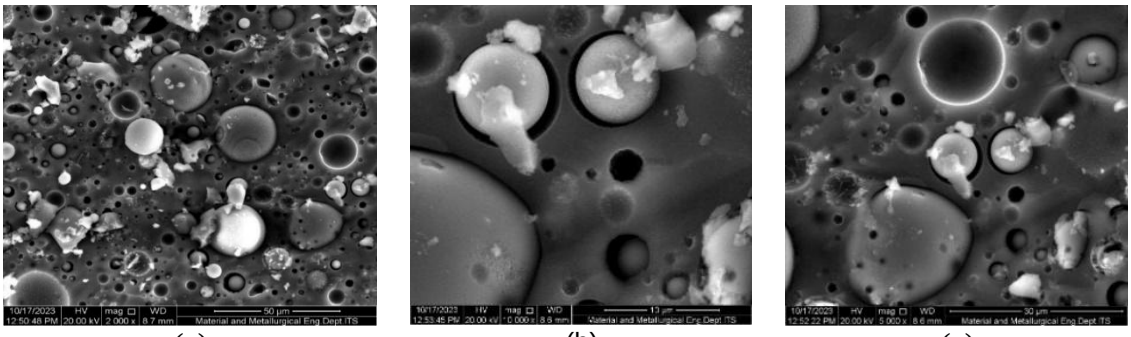
The *geopolymer's compressive strength and split tensile strength are influenced more by the amorphous Si and Al in the reactivity* than by that in the crystal phase [97], [98]. Due to the brittle nature of the crystal phase, *geopolymers* with higher crystal phase content exhibit lower *compressive strength* [99]. *Geopolymers* with Si and Al in an amorphous phase are more

active than those in a crystal phase, affecting the *compressive strength* and *split tensile strength* [71].

The room of application of the *geopolymer* depends on the effect of pineapple peel pectin addition on the increase of *compressive strength* and *split tensile strength* [100], [101]. Figures 3 and 4 compare the data of various types of *geopolymer* prepared with pectin.

Table 3. Comparison of Data Synthesis Geopolymer.

Variation	Wavelength (cm^{-1})	Functional Group	2θ (°)	crystal phase	Structural Characteristics
0%	3425.58, 1002.98, 1419.61	-OH stretching, HOH stretching, OCO stretching (Na_2CO_3)	20.90, 26.68, 50.20	Quartz (SiO_2), Magnetite (Fe_3O_4), Mullite ($3Al_2O_3 \cdot 2SiO_2$), Potassium (K), Molybdite (MoO_3)	The crystalline phase is more dominant than other variations
1 %	3425.58, 1018.41, 1419.61	-OH stretching, HOH stretching, OCO stretching (Na_2CO_3)	20.90, 26.68, 50.20	Dominance of Quartz (SiO_2), Mullite, and Amorphous is more extensive	It has a more significant and broader amorphous structure than other variations and the highest compressive strength.
2,5 %	3448.72, 1002.98, 1419.61	-OH stretching, HOH stretching, OCO stretching (Na_2CO_3)	20.90, 26.68, 50.20	Quartz (SiO_2), Mullite, and Amorphous are more dominant than 0%	Higher intensity, but can reduce the compressive and tensile strength
Pektin	3433.29, 1350.17	-OH stretching, -CH stretching			Having functional groups linked by hydrogen and carbon bonds



(a) (b) (c)

Figure 5. Micrograph of SEM results with magnification (a) 2000x, (b) 5000x, and (iii) 10,000x with variations in 1% addition of pineapple peel pectin on the compressive strength of geopolymer.

This experiment used the *geopolymer* sample added with pineapple peel pectin at the highest *compressive strength*, i.e., 1% variation. The magnifications of the SEM instrument used to obtain the morphologies of the *geopolymer* samples are presented in Figure 5: 2000×, 5000×, and 10,000× (variation at 1% in the

employed pineapple peel pectin). Coal fly ash and some unreacted materials are present in the *geopolymer* sample containing 1% added pineapple peel pectin variation (Figure 5b). This unreacted material will affect the *compressive strength* and *split tensile strength* of the *geopolymers* [42], [95], [100].

The 1% pectin variation has dominated the geopolymer pore formation, affecting the geopolymer's compressive strength and split tensile strength. The *geopolymer* pores are caused by trapped air bubbles in the paste due to imperfect vibration processing [42]. An instance of a needle-like structure appearing on the surface of the *geopolymer* paste is given in Figure 5.

Another cause of the needle structure is the high concentration of the *alkali solution*, or vice versa. After the *polymerisation* reaction occurs, the *alkali solution* does not fully react, resulting in needle structure formation [102]. For the *geopolymer* samples observed under SEM with and without 1% pectin variation [41], [103], because of the non-reactive pectin with the *geopolymer* matrix, the pectin appears inhomogeneous in Figure 5.

The varied quantity of pineapple peel pectin leads to differences in the surface area of *geopolymer* test samples, affecting the increases in *compressive strength* and *split tensile strength* of the *geopolymer* [100], [101]. The variation of the data points for the pectin types during *geopolymer* preparation is described based on Figures 3 and 4.

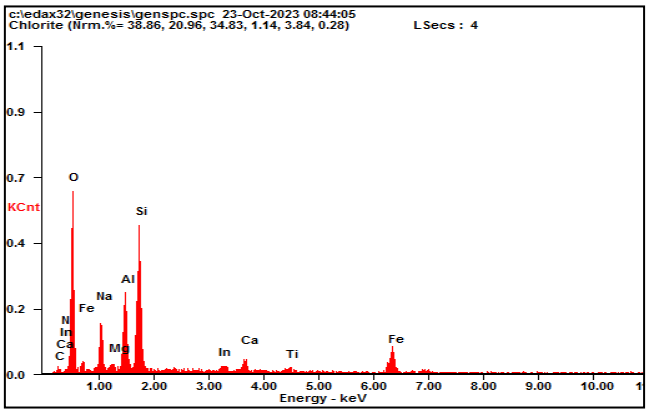


Figure 6. EDX (*Energy Dispersive X-Ray)* results of geopolymer with adding 1% pineapple peel pectin.

The *EDX* analysis of the composition corresponds well to the results of *XRD* and *XRF*, which demonstrated the inorganic nature of the components of the *geopolymeric* matrix: silicon (Si) and aluminum (Al). According to XRD results, Silicon is present from the SiO_2 compound in the mineral (Q) quartz. Mullite, with the formula (M) $3Al_2O_3{\cdot}2SiO_2$, contains aluminum and oxygen. The Felement originates from the mineral (Ma) magnetite (Fe_3O_4) originating from fly ash of Tanjung Jati B Jepara coal [104]. Element C is due to pectin content in the *geopolymer* sample [104]. Na is contained in the *XRF* test results, one of the substances derived from coal fly

ash and the *alkali activator* solution (NaOH and Na_2SiO_3).

(a) (b) (c) (d)

Figure 7. Compressive strength results of geopolymer with the addition of pineapple peel pectin (a). Variation 0%, (b). Variation 1%. Results of tensile strength (c). Variation 0% (d). Variation: 1% pineapple peel pectin

Figure 7 shows *geopolymer* containing pineapple peel pectin under different ages of 28 days for *compressive strength* and *split tensile strength*. The *geopolymer's compressive and split tensile strengths* increased by using pineapple peel pectin, but decreased after adding pectin at more than 1%. A pineapple peel pectin content greater than 1% causes the *compressive strength* and *split tensile strength* to reduce. Pineapple peel pectin is incorporated to retard the *geopolymer's* setting time and to increase the *viscosity* of the *geopolymer* paste, which influences the *compressive strength* and *split tensile strength* of the *geopolymer* [105]. As reported in previous research, the higher the measured *viscosity* value, the higher the *compressive strength* of the *geopolymer* [105].

The reduction in *compressive strength* and *split tensile strength* in the 2.5% *geopolymer* variation when added with pineapple peel pectin is due to excessive pectin disturbing the hardening time, which becomes too long. *XRD* results suggest that the aluminosilicate gel (N-A-S-H/C-A-S-H) formation intensity decreases with increasing pectin content, indicating incomplete *polymerization* due to organic compound interference. A surplus of hydroxyl (–OH) and carboxyl (–COOH) groups from pectin can compete with silicate species, changing the reaction kinetics and causing differences in the *geopolymer* matrix [85], [87].

The excessive dosage of pectin also causes cracks in the *geopolymer*: the *compressive strength* and *split tensile strength* decrease due to adding 2.5% pectin. The *compressive strength* and *split tensile strength* in the 1% variation with pineapple peel pectin were not achieved at optimum values, as pores were still present.

According to chemical and physical examination results, adding pineapple peel pectin influences the increase in compressive strength and split tensile strength of the geopolymer, but the values are not optimal. The sample with 1% pineapple peel pectin achieved the highest *compressive strength* in this study, 22.13 MPa; thus, it is categorized as medium quality concrete. For structural work, medium-strength concrete typically has a *compressive strength* in <K250 g/cm³ – <K400 g/cm³ or approximately 20 MPa – 35 MPa (ACI 318).

Regarding the properties of *geopolymers*, this research highlights certain limitations and proposes the need for further research. The influence of pectin on the durability of *geopolymers* should be evaluated in terms of exposure to moisture, temperature, and aggressive chemicals. In practical applications, selecting the appropriate pectin content is significant for optimizing material performance and cost-effectiveness for industry or construction use [87], [88], [106]. Potential applications include eco-friendly precast components, lightweight wall panels, and mortar for sustainable structures where balancing strength, hardening time, and environmental resistance is crucial. Nevertheless, long-term durability studies are necessary to evaluate resistance to moisture, chemical attacks, and freeze–thaw cycles.

CONCLUSION

Based on the results and discussion that have been done, it can be concluded that the presence of pectin from pineapple skin in the synthesis of *geopolymer* based on *X-ray Diffraction* (*XRD*) only provides evidence that the *geopolymer* contains an amorphous phase as well as quartz as a primary mineral, indicated by a hump in 2θ in the range of 15°–40°. The functional group analysis by *Fourier Transform Infrared* (*FTIR*) showed that CH bonds and carboxyl groups (–C=O) were formed in the *geopolymer* with 1% and 2.5% variations of pineapple skin pectin addition. In the morphological analysis with *Scanning Electron Microscopy–Energy Dispersive X-ray* (*SEM-EDX*) at a 1% pectin addition variation, the *geopolymer* containing pineapple peel pectin displayed a less homogeneous matrix. However, visible surfaces and unreacted materials remained. In the *geopolymer* sample with 1% variation of pectin addition, the distribution of pineapple peel pectin was still not uniform (inhomogeneous), but no visible cracks appeared. Furthermore, the *EDX* surface analysis results matched the *XRF* (*X-ray Fluorescence*) and *XRD* results.

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