

## Proximity Porosity and Crystallinity Analysis as Clay/Nickel Slag Characteristics for Material Stabilization Application

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Received: 17 January 2024, Revised: 18 February 2024, Accepted: 25 February 2024, Published: 10 June 2024

### Abstract

Slag nikel is a by-product of nickel ore smelting through the pyrometallurgical process, which contains silica (> 50 %). This suggests that nickel slag, possessing pozzolanic properties, which potentially to be used as a binding material in construction projects dealing with low soil bearing capacity, such as soil improvement for road foundation. Pozzolanic reactions are greatly influenced by the chemical bonds formed between the binding material and the soil, causing mechanical characteristic changes in the soil. This study aims to examine the effect of adding nickel slag to soft soil based on changes in its physical, mechanical and chemical properties. Physical and mechanical property tests were conducted according to ASTM standards, while the changes in chemical structure in the soil were analyzed based on X-Ray Diffraction (XRD) test results. In this study, nickel slag percentages of 3, 6, 9 and 12 % were used based on the weight ratio of clay soil, at optimum water content ( $w_{opt}$ ). Furthermore, the test specimens were cured for 14, 28 and 56 days to observe the effect of time on the increase of crystal phases in each variation. The results showed that the presence of nickel slag in the soil can affect its physical and mechanical properties. The nickel slag content in the soil of 3, 6, 9 and 12 % reduces the plasticity index by 16.69, 12.02, 8.86 and 6.38 %, respectively. Meanwhile, the density values increased by 10.97, 11.27, 11.68 and 12.01 kN/m<sup>3</sup>, respectively. The changes in physical and mechanical properties can be explained by the XRD analysis results, showing changes in the spectrum curve and peak intensity of X-Ray Diffraction in the soil with the addition of nickel slag. This explanation clarifies the transformation of the soil's atomic structure from an amorphous state to a crystalline form, which is stabilized by nickel slag.

**Keywords:** Nickel slag, soft soil stabilization, XRD analysis

### Introduction

The extent of soft clays in Indonesia reaches 10 % of its territory or 20 million hectares [1]. Soft clays are highly susceptible to volume changes that are directly related to changes in moisture content. This is due to the presence of clay minerals and their ability to retain moisture. These physical properties explain the low bearing capacity of soft clays [2]. Mitigating the impact of soft clay behavior on infrastructure development has become a major challenge for geotechnical engineers, particularly in road foundation structure. The use of chemicals as stabilization materials has been widely used and can have a positive effect on improving the mechanical properties of soft clays in terms of strength and hardness.

Cement and lime are conventional materials that have been widely accepted as stabilization materials in soft clays [3]. In the production of 1 ton of cement, it can generate carbon dioxide gas of 0.73 - 0.85 tons [4], while the production of 1 ton of lime produces 0.32 tons of CO<sub>2</sub> [5]. The high demand for both of these materials in construction will certainly have an impact on environmental damage, especially greenhouse gas effects. Material engineering that utilizes industrial by-products becomes an alternative, aiming not only to increase the mechanical value of the material but also to enhance the economic value of waste and reduce the environmental damage caused by CO<sub>2</sub> emissions [6].

North Maluku is one of the largest nickel producers in Indonesia with potential nickel deposits reaching 1.4 billion tons [7]. Currently, the nickel ore processing industry in North Maluku continues to grow in line with the increasing demand for nickel, both nationally and globally. The nickel ore processing process produces by-products as much as 50 times the nickel ore produced, where one of the residual materials is nickel slag. Nickel slag produced from the nickel industry on Obi island has a mineral content of SiO<sub>2</sub> of 44.89 %, Fe<sub>2</sub>O<sub>3</sub> of 25.11 %, MgO of 20.27 % and CaO of 3.34 % [8]. The mineral composition of this nickel slag indicates that this material is pozzolanic in nature which can be used as a stabilization material in clay.

In principle, chemical stabilization efforts can reduce clay pores through the bonding of binder particles with clay material [9]. The decrease in clay porosity occurs in line with the treatment period of the mixed material which results in a reduction in the pore number and permeability value of the clay. This is why chemical stabilization efforts have a higher resistance and significant increase in clay bearing capacity [10,11]. This clay cementation mechanism will then increase the density and strength of the clay in accepting loads, thus meeting technical requirements, especially in the construction of road foundation layers [12]. The primary factor contributing to this is its cost-effectiveness and significant stability, both in the short and long term [13].

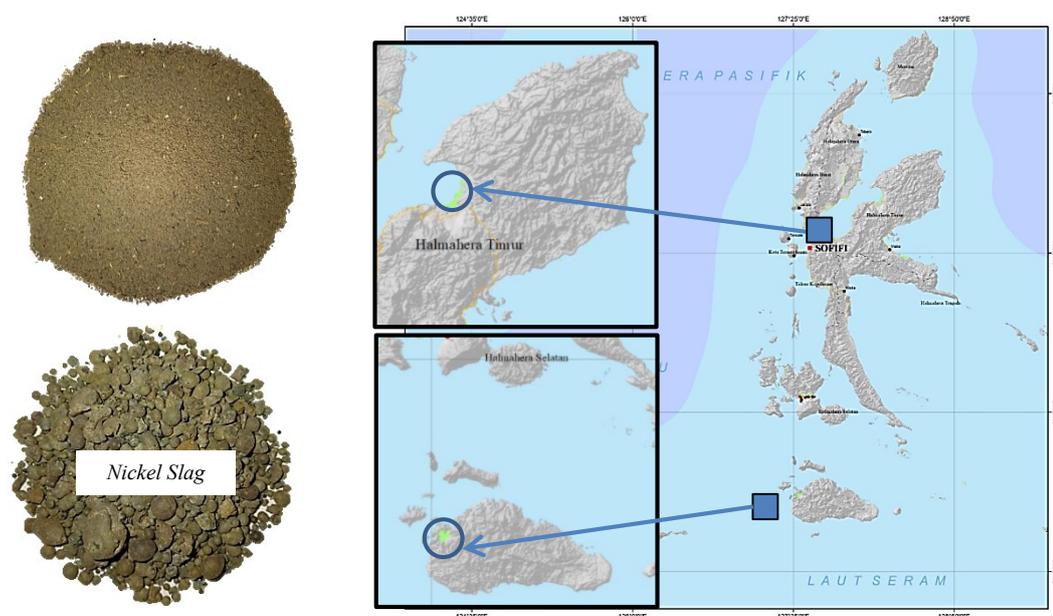
In chemical stabilization efforts, many studies have explained that the chemical components of stabilization materials greatly affect the mechanical value of soft clays [14,15]. This is because the chemical bonds that occur between clay minerals and binding materials strengthen the structure of the material from within [16]. The enhancement of the bearing capacity of clay in clay cementation occurs through pozzolanic and hydration reactions, through the reaction between calcium hydration of alumina and/or silica to water. The end result of this reaction is highly dependent on the presence of silica and/or alumina minerals, which can be calcium silica hydration and/or calcium alumina hydration [17]. Thus, understanding the changes in chemical characteristics of stabilized clays is very important to assess their mechanical characteristics. As for the efforts that can be made to understand the shape changes and chemical bonding in clay-cement, X-Ray Diffraction (XRD) testing is an excellent tool to see the crystal phase changes in solid materials [18].

The concept of crystallinity is related to the quantification of the structural organization inherent in solids [19]. The crystalline characteristics of an object show a remarkable regularity of mineralogical structure, characterized by the presence of periodically arranged atoms and molecules. The phenomenon of crystallinity exerts a visible influence on various material properties, including but not limited to hardness, density, transparency and diffusion [19]. In geopolymers, the degree of crystallinity increases as the regularity of the geopolymer chains increases and with fewer short branches, which allows the molecules to gather close together.

## Materials and methods

### Material sampling quarry

The clay used in this study was taken from Subaim village, which is administratively located in East Halmahera Regency, North Maluku Province. This is based on the Atlas of Soft Clay Distribution showing the presence of soft clay found in the Subaim bay area [20]. The nickel slag used as stabilization material in this study was obtained from the nickel ore processing industry located on Obi Island, which is administratively located in South Halmahera Regency. The collection location and visual form of the clay and slag materials are shown in **Figure 1**.



**Figure 1** Soft clay and nickel slag quarry.

### Samples preparation

XRD test material samples in this study were obtained from the results of laboratory compaction testing. The composition of clay and nickel slag mixture is based on weight ratio, where the percentage of nickel used is 3, 6, 9 and 12 %. Before being applied as a stabilization material, nickel slag granules must go through a smoothing stage using a grinding machine. This process is necessary to obtain fine and homogeneous grains, while the slag material used is material that passes sieve No. 200. This is based on the opinion that the reactivation of stabilization materials is strongly influenced by the level of fineness of the grains, otherwise known as the specific surface area [21]. Thus, the higher the specific surface, the faster the material reacts.

The initial stage of this research was carried out by testing the physical properties of soft clay used to determine the type of soft clay used. All tests of clay physical properties refer to ASTM standards, including: Sieve Analysis (ASTM D6913), Specific Gravity (ASTM D854), Water Content (ASTM D2216) and Atterberg Limits (ASTM D4318). After the physical properties of the clay were obtained, mechanical properties testing was conducted where only laboratory compaction testing was conducted.

The procedure for making compaction test specimens in this study was carried out by referring to the ASTM D698 standard. The clay material taken from the location was dried in the sun until air drained, after which the clay was sieved and the clay material used for the manufacture of compaction samples was clay that passed the No. 4 sieve. The mixing of clay and nickel slag was carried out in a dry state and matured for 24 h. The making of test specimens is done by mixing water into the mixture matrix by adding water little by little and stirring for 10 min until the mixture is homogeneous [22]. From the results of the compaction test, samples of clay matrix and nickel slag at optimum moisture content of 15 - 25 g were selected as test materials for XRD analysis.

### Material characterization testing

X-ray Diffraction (XRD) testing is an important tool in the study of mineralogical materials. Measurements and analyses using X-ray Diffraction (XRD) techniques are intended to identify, quantify, determine the type of mineral, chemical composition, crystal structure and other physical and chemical properties of minerals from complex mineral combinations [23]. In this study, XRD testing was carried out at the Physics Laboratory, Hasanuddin University. This measurement is performed using X-ray diffractometer SHIMADZU 7000 with  $\text{CuK}\alpha$  radiation ( $\lambda_{\text{Cu}} = 1.54 \text{ \AA}$ ) in the range ( $2\theta = 10 - 70^\circ$ ), operation 30 kV and 10 mA [24]. The diffraction measurement results of the test samples were then analyzed using Match and Maud software followed step from our previous study [25,26]. The principle of this application is to present the diffraction pattern based on the X-ray intensity at an angle of  $2\theta$ , which is then matched with the distribution pattern formed that have been stored in the international data base.

## Results and discussion

### Physical characteristics of nickel slag stabilized clay

The test results of the physical properties of soft clay used in this study are shown in **Table 1**. From the sieve analysis results, the clay percentage of 78 % indicates that the type of clay used in this study is a fine-grained clay. In fine-grained clays, plastic properties become an important parameter in construction, because it is related to the adaptability to constant changes in shape and volume [27]. Based on the USCS clay classification, the soft clay used in this study can be classified as organic clay with high plasticity (OH). This is based on the relationship between the plasticity index (IP) value of 21.73 % and the liquid limit (LL) value of 63.92 %, which is plotted on the Casagrande Clay Classification Diagram, as shown in **Figure 2**.

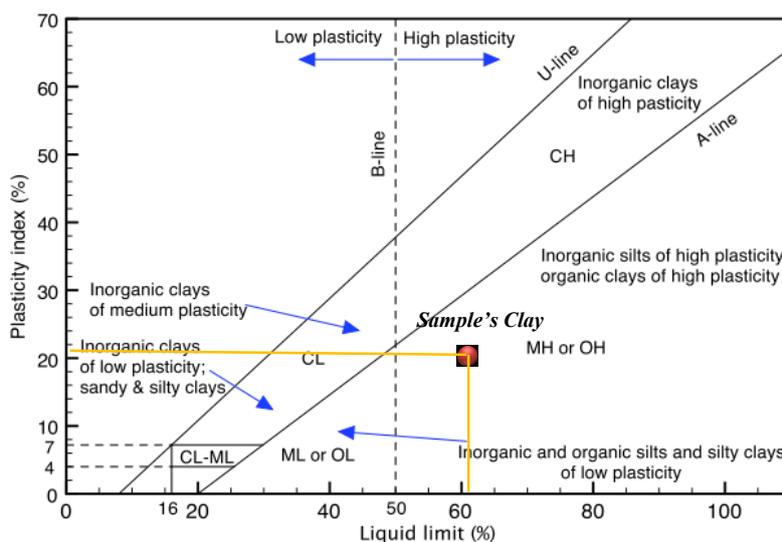
The behavior of soil stabilized chemically can be demonstrated through its physical changes, wherein in this study, these changes are observed based on the Atterberg limit values. Based on the test results, the Atterberg limit values due to the addition of nickel slag to soft soil are shown in **Table 2**. In general, the test results indicate that the higher the content of added nickel slag, the lower the liquid limit (LL) and the higher the plastic limit (PL) and plasticity index (PI) values of the material. **Table 1** shows that the addition of nickel slag can reduce the liquid limit (LL) by 3.46 - 11.64 %, while the plastic limit (PL) value increases by 3.45 - 7.96 %. Changes in LL and PL values certainly affect the plasticity index (IP) of the clay, where the percentage of nickel slag will reduce the plasticity of soft soil from high plasticity ( $\text{PI} > 17\%$ ) to medium plasticity ( $7 < \text{PI} < 17$ ).

**Table 1** Properties of soft clay.

Physical characteristics	Value
Specific gravity (Gs)	1.64
Optimum moisture content ( $w_{opt}$ , %)	36.08
Sieve analysis	
Sand (%)	8
Silt (%)	14
Clay (%)	78
Atterberg limits	
Liquid Limit (LL)	63.92
Plastic Limit (PL)	42.16
Plasticity Index (PI)	21.73

**Table 2** Atterberg limit parameters of soft clay stabilized by nickel slag.

Physical Characteristics	Value				
	0 %	3 %	6 %	9 %	12 %
Atterberg Limits					
Liquid Limit (LL)	63.92	61.63	59.31	57.25	54.26
Plastic Limit (PL)	42.16	44.95	47.29	48.13	48.13
Plasticity Index (PI)	21.73	16.69	12.02	8.86	6.38



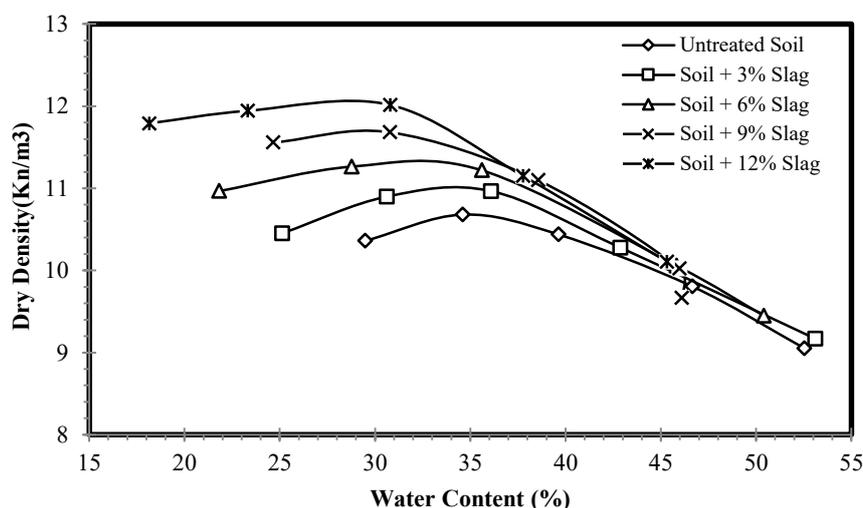
**Figure 2** Clay classification based on USCS.

**Density characteristics of nickel slag stabilized soft clays**

In this research, testing the mechanical properties of clay is seen based on the density value or volume weight of the clay. This test refers to the ASTM D698 Standard. The results of the compaction test are shown in **Figure 3**. From this graph, it can be seen that in general there is an increase in the clay volume

weight value along with the increase in the percentage of nickel slag in the clay, where the increase varies between 2.66 - 12.57 %, with the optimum moisture content in the interval 30 - 36 %.

The results showed that the dry density characteristics ( $\gamma_{dry}$ ) of the clay and nickel slag mixture matrix continued to increase, where for nickel slag levels of 3, 6, 9 and 12 % produced dry density values ( $\gamma_{dry}$ ) of 10.97, 11.27, 11.68 and 12.01 kN/m<sup>3</sup>, respectively. This increase is in line with the results of the clay physical test, where nickel slag is able to reduce the absorption of moisture content in clay minerals. The density of clay particles will then affect the permeability of the clay, where the larger the pore space in the clay, the easier it is for water to continue to flow following gravity.



**Figure 3** Relation of water content with dry density.

#### Chemical compounds of clay

Mineral characterization was carried out through X-Ray Diffraction (XRD) testing to determine the type of minerals and estimate the percentage of clay minerals. The results of XRD testing of clay samples are in the form of a relationship between intensity and diffraction angle ( $2\theta$ ). The mineral characterization process was continued by matching the diffraction peaks. The matching of diffraction peaks was carried out using the Match! 2.0 programme based on the XRD spectra as input. Based on XRD analysis, the soft clay in this study mostly consists of amorphous and crystallinity index 90.91 and 9.01 %, respectively, as shown in **Figure 4(a)**. The spectrum pattern formed by this measurement can explain that the clay used is amorphous. This is indicated by the ratio between silica/alumina which is smaller than 1 [28]. Based on the search and match results, the mineral content and percentage of the clay samples can be identified which include: Smectite and Halloysite 53.90 %, Illite 22.80 % and Quartz 14.50 %. Thus amorphous clay-size materials formed under that environment typically have a silica/alumina ratio of less than 1 [28]. This type of amorphous clay-size materials can be characterized as alumina-rich. The presence of gibbsite and the absence of smectite in these clays are indications of the highly leached, silica-deficient, alumina-rich clay environments.

We also detect the Fayalite as dominant phase for nickel slag sample which has good correlation to the Refs [29,30]. Fayalite is an orthorhombic crystalline material, which consists of SiO<sub>2</sub> with a tetrahedral structure, which is formed by a Si atom at the center coordinate and 4 O atoms binding it. Olivine-type silicate, Fe atoms are connected together with SiO<sub>4</sub> tetrahedral, and 6 O atoms surround the Fe atoms. Under these conditions Fayalite has a tendency as a semiconductor's properties with such a combination, nickel slag will provide density to the sample if used as doping so that it will have a linear impact on its LL and PL.

#### Chemical compounds of nickel slag

The results of the mineralogical characterization of nickel slag as a doping for clay matrix are shown in **Figure 4(b)**. The spectrum of nickel slag produced 24 identifiable peaks. The highest peaks were recorded at theta angles of 52, 36 and 32 ° indicating the presence of Fayalite and magnetite minerals that suggested from nickel slag, and kaolinite mineral from clay. The high presence of silica in this sample indicates that nickel slag has the potential to be used as a stabilization material in soft clays. The Miller

index is obtained and expressed in fields (hkl): (020), (110), (021), (100), (111), (130), (112), (200), (113), (222), (004), (114) and (201). The enhanced spectra confirmed that amorphous index had been converted using the Segal method. The amorphous part can be assigned as a hill in the range of 15 - 70 ° on the XRD curve in degrees 2θ by using the following equation ( $X = X_c + X_a$ ), where X is total of crystalline ( $X_c$ ) and amorphous ( $X_a$ ) index.

$$X = pI_c + qI_a \quad (1)$$

$$qI_a = X - pI_c \text{ and } I_a = \frac{X}{q} - p \frac{I_c}{q} \quad (2)$$

$$C_r = \frac{X_c}{X} 100 \% ; \text{ or } C_r = \frac{I_c}{I_c + \frac{qI_a}{p}} 100 \% ; p \text{ and } q \text{ as proportional constant} \quad (3)$$

This calculation uses peak intensities and any substantial inaccuracies in the calculation can be affected by grain size irregularity orientation. The porosity calculation starts by considering the Fayalite lattice parameter as dominant phase on the clay/nickel slag sample which can be expressed by [31], as presented below:

$$\frac{1}{d^2} = \frac{1}{v^2} (S_{11}h^2 + S_{22}k^2 + S_{33}l^2 + 2S_{12}hk + 2S_{23}kl + 2S_{13}hl) \quad (4)$$

where,

$$S_{11} = b^2c^2\sin^2(\alpha), \quad (5)$$

$$S_{22} = a^2c^2\sin^2(\beta), \quad (6)$$

$$S_{33} = a^2b^2\sin^2(\gamma), \quad (7)$$

$$S_{12} = abc^2(\cos(\alpha)\cos(\beta) - \cos(\gamma)), \quad (8)$$

$$S_{12} = abc^2(\cos(\beta)\cos(\gamma) - \cos(\alpha)), \quad (9)$$

$$S_{12} = abc^2(\cos(\gamma)\cos(\alpha) - \cos(\beta)), \quad (10)$$

We have to note that triclinic substances have great complexity caused by the number of independent constants ( $a \neq b \neq c$ ), respectively, so the Eq. (4) is approximately to find the most probable parameter value. Then volume (V) of unit cell ( $\alpha \neq \beta \neq \gamma \neq 90^\circ$ ) following equation:

$$V_{\text{triclinic}} = \sqrt{1 - \cos(\alpha)^2 - \cos(\beta)^2 - \cos(\gamma)^2 + 2 \times \cos(\alpha) \times \cos(\beta) \times \cos(\gamma)} \quad (11)$$

The formula to determine the porosity can be expressed:

$$P = \left(1 - \frac{\rho_{\text{ex}}}{\rho_s}\right) \times 100 \% \quad (12)$$

where the x-ray density ( $\rho_s$ ), and the experimental density ( $\rho_{\text{ex}}$ ) are extracted from:

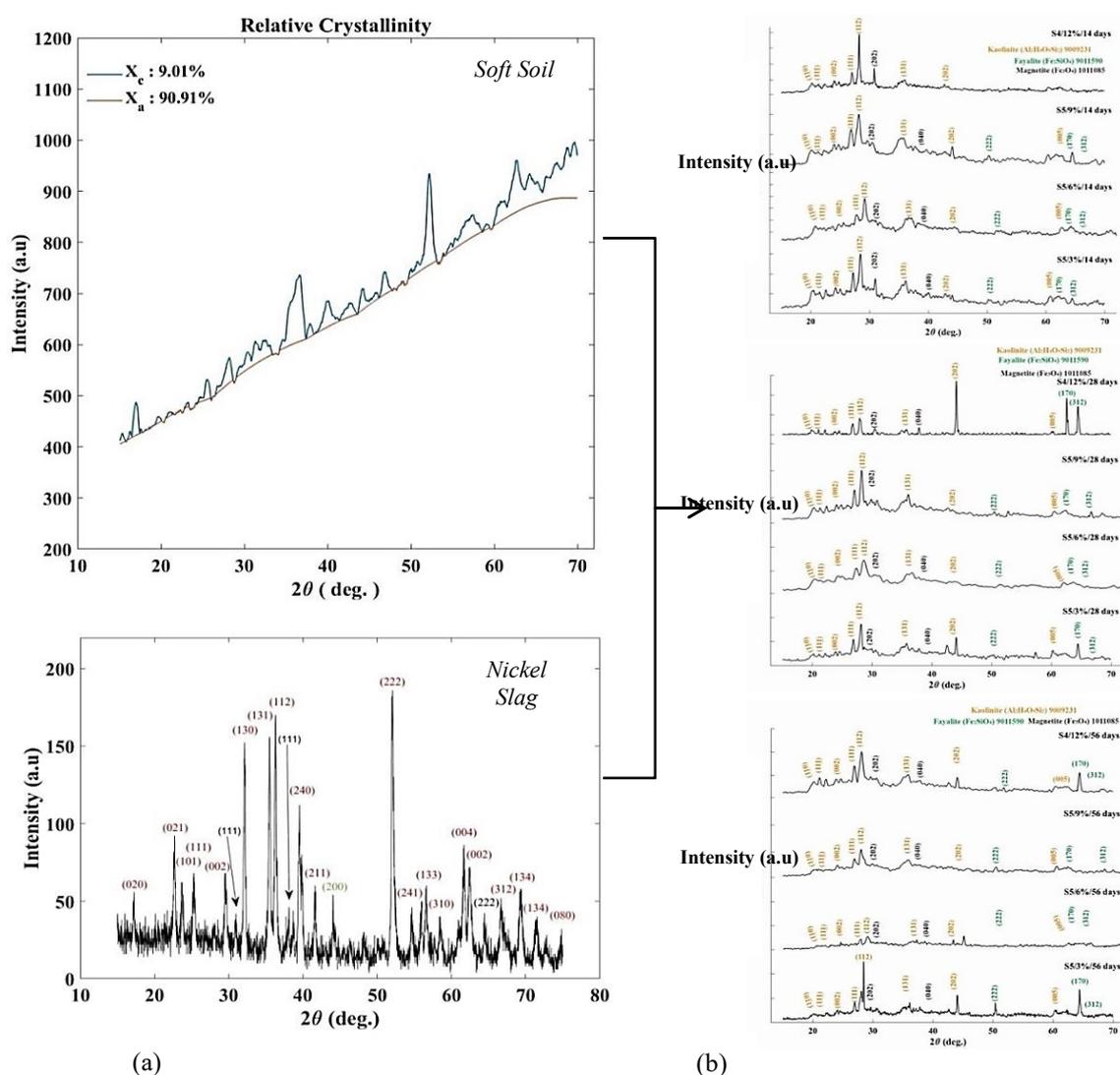
$$\rho_s = \frac{nM}{NV_{\text{triclinic}}} \text{ and } \rho_{\text{ex}} = \frac{m}{V_{\text{exp}}} \quad (13)$$

Number of molecules per unit cell (n) for weighted values for  $\text{Fe}_2\text{SiO}_4$ , molecular weight (M) and Avogadro's number (N). The from pellet from XRD preparation, we can measure the mass (m) and volume (V) of the sample.

#### Characterization of clay stabilized by nickel slag

The physical and mechanical behaviors of nickel stabilized clay in this study was validated based on the results of X-Ray Diffraction testing to see changes in nickel slag clay compounds. The XRD test results

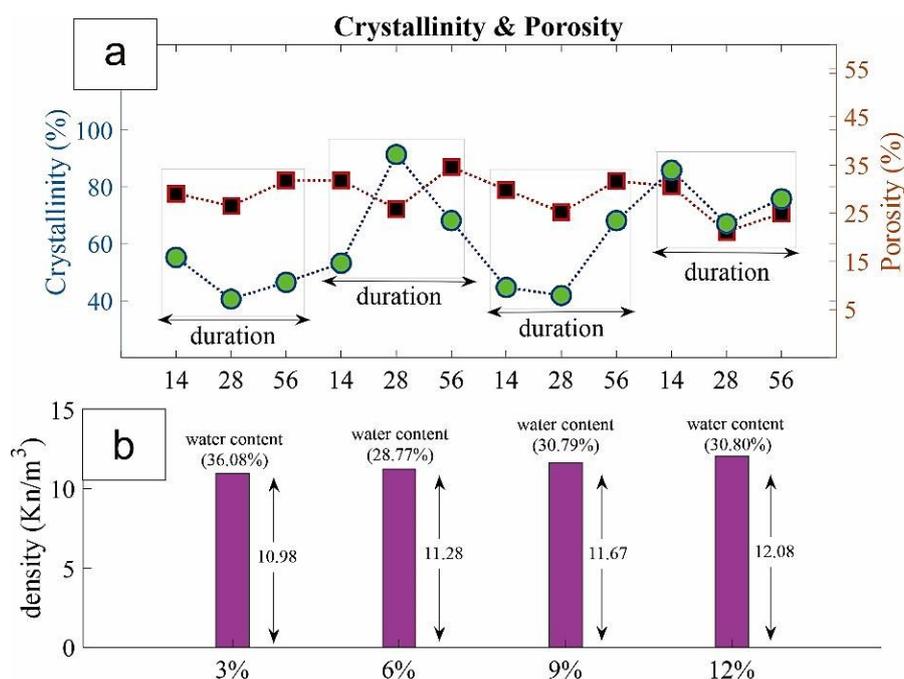
on the clay matrix with varying levels of nickel slag are shown in **Figure 5**. Based on the graph (**Figure 5(a)**), the amorphous phase and porosity has the role that influence the density and water content to the sample. It is found that, the highest water content and density is shown by clay with nickel slag (12 %) which can be influenced by the close proximity of porosity and crystallinity. Further, based on **Figure 5(b)**, it is found that the lowest water content is shown by clay with nickel slag (6 %), the lowest density is shown by clay/nickel slag (3 %), which can be influenced by the inconsistency proximity of porosity and crystallinity. Based on these results, it can be stated that structural properties parameters such as porosity and crystallinity can determine the density and hydrophobicity of the material [32,33]. Clay density is also influenced by its mineral phases such as illite, kaolinite, quartz, calcite and dolomite which have a reactive external response to moisture. Where the addition of nickel slag in this study managed to significantly compact the clay with the phenomenon of crystalline phase change so that it is not reactive to external chemical processes.



**Figure 4** (a) Diffraction spectra of nickel slag and clay and (b) composite of clay/nickel slag with different concentration ( $x = 3, 6, 9$  and  $12\%$ ).

As evidenced by XRD spectrum analysis, the presence of nickel slag in the soil matrix modifies the diffraction intensity pattern as shown in **Figure 4(b)**. The improvement of the diffraction pattern in the clay matrix can be clearly seen, where the diffraction pattern that previously experienced a steadily increasing baseline pattern such as a slope becomes a normal diffraction pattern after the addition of nickel slag. The increasing trend observed in the baseline pattern indicates natural changes in the clay, and the presence of

nickel slag normalizes the pattern, which will help in analyzing the effectiveness of an external intervention on the sample [33]. Variations in density and moisture content as a function of nickel slag concentration are also due to the unique properties of the individual components and their interactions [34]. Particle packing and porosity are influences that are affected by shape, and different packing characteristics. The addition of nickel slag, which may have a different particle size and shape distribution compared to clay, can affect the overall packing of particles in the mix. Water Retention Clay minerals that are known for their ability to absorb and retain water due to their high surface area and cation exchange capacity are also parameters that led to the modification of our sample, but keep in mind that nickel slag, on the other hand, may not have the same water absorption characteristics [35]. The combination of these materials can affect the overall water absorption and retention properties of the mix, affecting the moisture content.



**Figure 5** (a) crystallinity and porosity as a function of duration and (b) water content and density as a function of nickel slag concentration (3, 6, 9 and 12 %).

## Conclusions

The objective of this research is to assess the potential for enhancing the geotechnical characteristics of organic soil through the chemical stabilization by utilization of nickel industry residue, specifically in the form of crushed nickel slag. The ongoing research project involves the testing of physical testing, mechanical testing and crystal phase analysis. Based on the acquired findings, several significant observations can be inferred that: The addition of nickel slag to soft soil affects the physical properties of the soil, with a decrease in the soil's plasticity index. This suggests that nickel slag is capable of reducing water absorption in the soil. The lower water content naturally affects its mechanical values, as the dry density of the soil material with added nickel slag experiences an increase. Changes in the physical and mechanical properties of the soil-nickel slag matrix can be explained based on XRD test results, where in the presence of nickel slag, the X-ray spectrum undergoes a transformation from a gradually rising baseline, resembling a slope, to a standard diffraction pattern. Therefore, it can be stated that, it can be concluded that the physical and mechanical characteristics are significantly influenced by the amorphous phase and material porosity. The results of this study are expected to be considered in efforts to chemically improve soil using nickel slag waste, which can be applied to the improvement of road foundation layers and the development of environmentally friendly bricks made from soft soil.

## Acknowledgment

We extend our sincere thanks to Khairun University for backing our research under the program *Penelitian Kompetitif Unggulan Perguruan Tinggi (PKUPT)*.

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