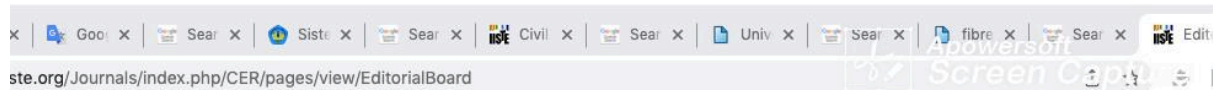


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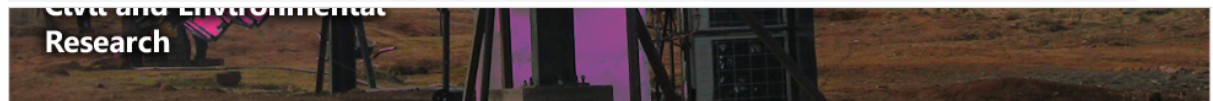
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The Evolution of Concrete (Part 3): Submerged in Mud and Its Compression Strength

Chatarina Niken*

Civil Engineering Department, Faculty of Engineering, Lampung University, Jl. Sumantri Brojonegoro No 1
Bandar Lampung, Indonesia

* E-mail of the corresponding author: chatarinaniken@yahoo.com

Abstract

This paper discusses the microstructural degradation mechanism of slurry submerged concrete and its compressive strength. This is based on an experimental study in the basement of the Lampung University Teaching Hospital in Indonesia. Samples were taken using four core drills in the basement under protected conditions and submerged in mud for compression testing. The core drill was taken for SEM and EDX testing. Compressive strength data is processed using ASTM E178-02 standard practice for dealing with outlying observation. EDX data for both types of concrete conditions that describe the dynamic viscosity of each chemical element are compared and linked with EDX data which states atomic mass and SEM. From this relationship, the mechanism of degradation of concrete submerged in mud is sought by studying literature. The results showed that the compressive strength of concrete was submerged in mud for 10 years, only 50% of the compressive strength of protected concrete. Degradation is caused by processes series of water infiltration, oxidation of the surface water that accompanies the immersion of the mud, immersion pressure which changes the atomic structure and density of Ca and the depletion of portlandite which is then followed by the release of Ca from CSH, the appearance of Fe^{3+} which makes structural changes, increased porosity. The right way to prevent concrete from degrading its strength is to make the concrete a minimum of water permeable and protect it with a waterproof layer.

Keywords: concrete, compressive strength, evolution, microstructure, submerged in mud

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1. Introduction

Concrete is widely used because of its excellent compressive strength, durability, and little maintenance. Concrete is a material that has undergone an interesting intellectual and practical renaissance in recent years (Goodbun 2016). The durability of concrete is determined by its inappropriate use or it can also be caused by the infiltration of the surrounding elements. The pore area of concrete is around $500m^2/cm^3$ (Bažant & Wittmann 1982), thus elements from outside are easy to infiltrate. Small soil particles, which are easy to infiltrate are clay and silt.

To protect the quality of hard concrete against the infiltration of these elements, for construction directly related to the ground, requires a 75mm concrete blanket (SNI 2847 2019). To maintain the quality of the hydration reaction in fresh concrete, silt, and clay in the aggregate as a building material for concrete a maximum of 5% (SNI 03-1750 1990). Studies on the effect of mud associated with concrete have been carried out by several researchers.

The use of sedimentary sludge in brick production has been studied (Pasri *et al.* 2021). The manufacture of lightweight concrete and cone blocks with Lapindo mud mixture has been carried out (DPUPKP 2020). Research on lightweight concrete from Lapindo, Sidoarjo, Indonesia mud as a structural concrete obtains a compressive strength that can reach a quality of f_c 20 MPa (K. 250) with a density of 1.3 – 1.4 kg/ltr where this value has met the requirements of SNI 2847-2019 concerning Requirements for Structural Concrete for Building (Lasino 2019).

The effect of different Lapindo mud preparations on the compound content has been studied (Ulfindrayani *et al.* 2019). Partial replacement of cement with Chikoko mud reduces the compressive strength of concrete (Ottos & Nyebuchi 2018). The compressive strength of red mud was found to be higher than conventional concrete up to 10% (Ramshad *et al.* 2018). The effect of sediment powder addition on compressive strength has been observed. (Nuhun *et al.* 2018). Mud sediment as a brick material has been studied (Ichsan 2018). Paving mud concrete blocks for pedestrian walkways has been observed (Udawattha 2017). The initial concept of developing mud-concrete was to incorporate both the strength and durability of concrete into mud-based constructions to introduce a low-cost, load-bearing wall system with easy construction techniques which ensured indoor comfort while minimizing the impact on the environment (Arooz & Halwatura 2017). The leaching performance of concrete based on studies of samples from old concrete construction has been published (Lagerblad 2001).

This infiltration can occur in structural elements that are in contact with the ground, or which due to natural conditions, buildings or structural elements become covered with soil. A concrete basement submerged in mud

also occurred on the basement floor of the PTN Hospital, University of Lampung, Indonesia. This happened from 2010 to 2020.

The degradation of concrete is usually known from its external appearance and its compressive strength. The outward appearance may look fine, but the microstructure has changed a lot. The study of the microstructure under degraded conditions due to infiltration of soil particles and the degradation process and its relationship to the compressive strength of concrete needs to be studied.

2. Materials and Methods

2.1. Materials

The PTN Hospital, University of Lampung, Indonesia which had been neglected since 2010-2020. The basement was submerged in water mixed with mud from the soil cliffs at the location for a period of 10 years. Samples were taken by core drill at locations that were submerged in mud and protected by a minimum of 4 samples each (Figure 1). The samples were used for compression testing, SEM (scanning electron microscopy), and EDX (energy dispersive X-ray).



Figure 1. Concrete condition: (a) Protected concrete, (b) Concrete submerged in mud

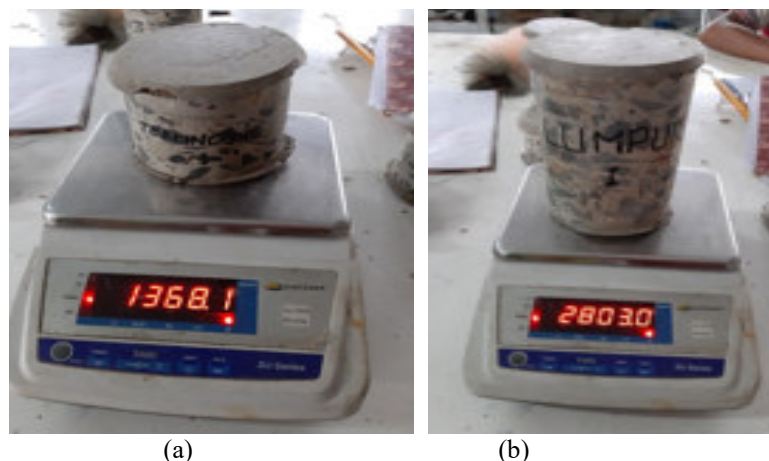


Figure 2. Core drill samples: (a) protected, (b) submerged in mud

2.2 Method

The results of the compression test were analyzed by calculating the standard deviation. The data is processed using ASTM E178-02 standard practice for dealing with outlying observation. There are 2 EDX test results, namely the relationship between dynamic viscosity - energy of the elements contained and their atomic masses. Each element and properties of the EDX results were studied from the literature, the effect of immersion in water mixed with mud on the mechanism of each element was analyzed.

There are four magnitudes used in the SEM test, namely 2.00K, 5.00K, 10.00K, and 15.00K. This is done so that the microstructure can be seen in its entirety. The relationship between the EDX results and the microstructure of the SEM test was analyzed between the protected and submerged conditions. EDX and SEM results were analyzed and integrated. The result is related to the compressive strength.

3. Result and Discussion

3.1. Result

3.1.1 Compression Test Result

From the analysis of ASTM E178-02 the results of the largest and smallest compressive strength of 4 samples of protected concrete and 4 samples of concrete submerged in mud are all below the 5% significant level so that all data can be accepted (Figure 3a). The compressive strength of concrete submerged in mud is 52% of the compressive strength of protected concrete (Figure 3b). Soaking mud makes the compressive strength of concrete only half of the compressive strength of the protected condition.

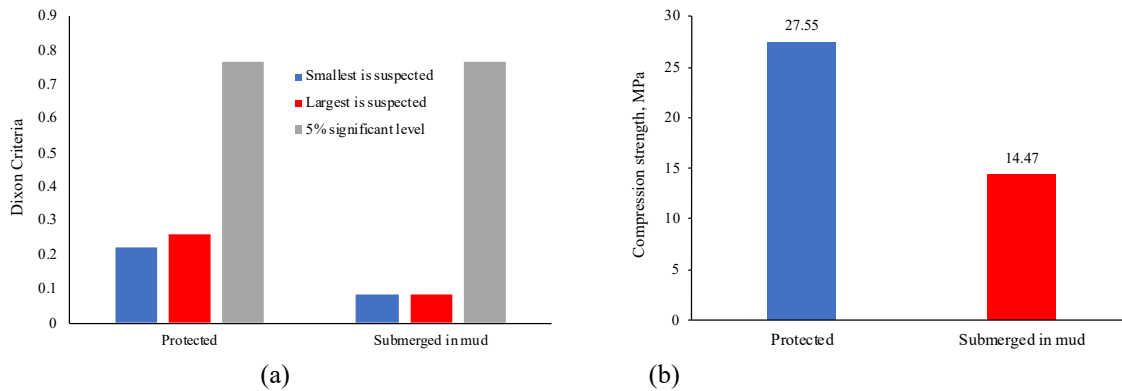


Figure 3. (a) Outlying analysis based on Dixon Criteria, (b) Compressive strength

3.1.2 Energy Dispersive X-ray Result

The results of the EDX test for protected and submerged conditions are presented in Figure 4. The ordinate is dynamic viscosity/cc (eps/ev), and absciss is energy.

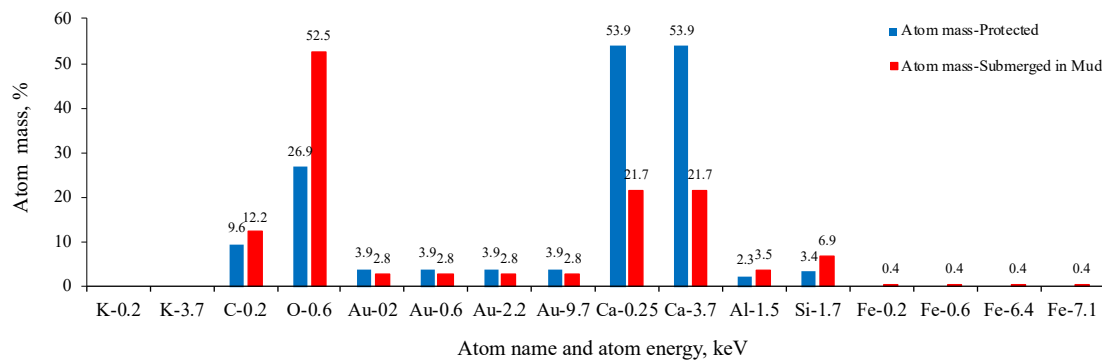


Figure 4. Relationship between Atom mass and atom energy

The atomic masses of O, Ca and Si have very contrasting differences. The atomic mass of O and Si in the mud submerged condition was almost twice that of the protected condition, while the atomic mass of Ca decreased by about $\pm 60\%$. Due to the entry of water, the solubility of Si in CSH and Ca in CH increases. This causes CSH and CH to become unstable, the atomic mass of Ca drops drastically. This makes concrete submerged in mud more heterogeneous. Its heterogeneous structure can cause negative effects (Uzbaş & Aydin 2019). The negative effect in this condition is the compressive strength drop from 27.55 MPa to 14.47 MPa. The longer the pores are full so that the Si does not move anymore and condense the surroundings, so that the atomic masses of C, O, Al and Si increase. Au is not able to be compacted by the action of Si, even the atomic mass of Au decreases (Figure 4).

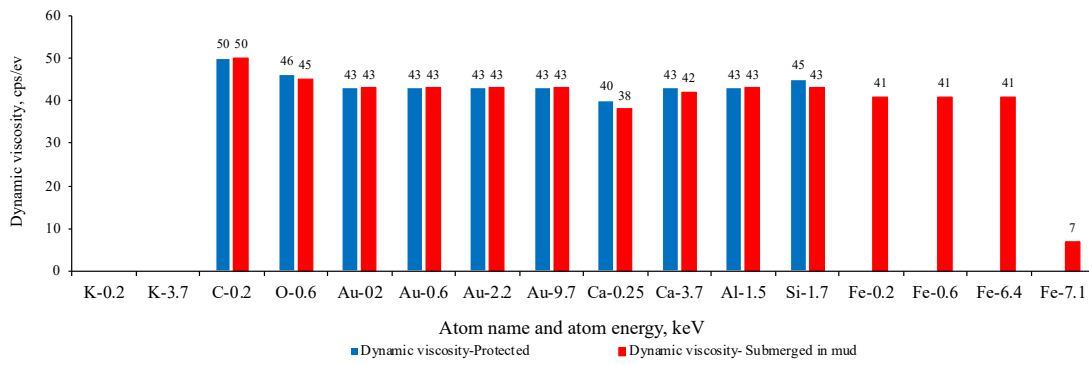


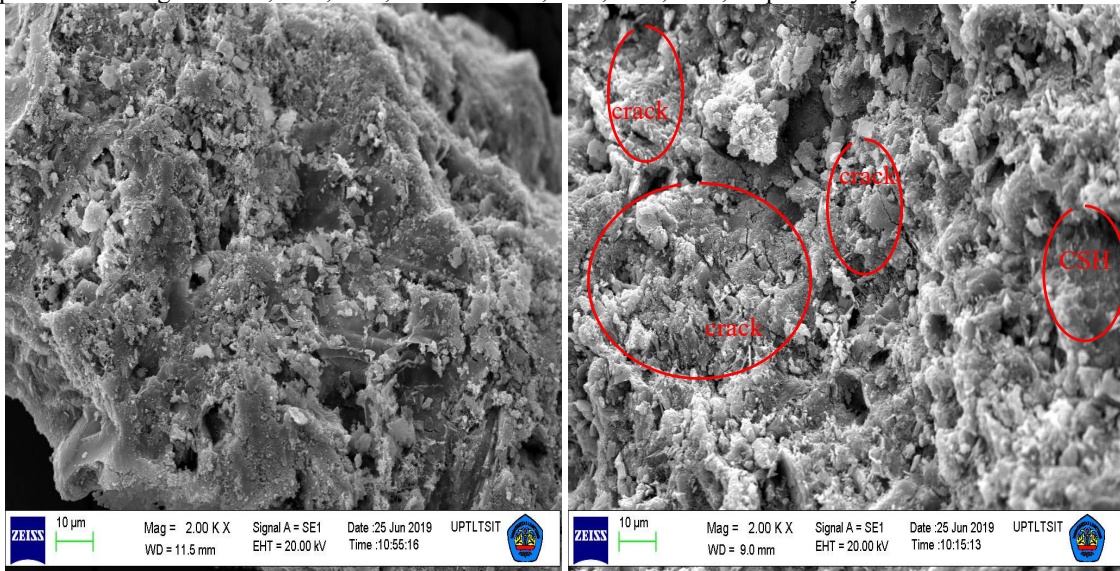
Figure 5. Relationship between dynamic viscosity and atom energy

There are similarities of dynamic viscosity and energy values from the EDX test of chemical elements of Ca, Au, O, C, Al, and Si in the protected and submerged samples (Figures 5). Fe element is not in the protected condition, but in concrete submerged in mud Fe appear with four types atom energy. This Fe is unstable and causes structural changes (Febrini *et al.* 2014). This structural change is shown in (Figure 6.1 to 6.4).

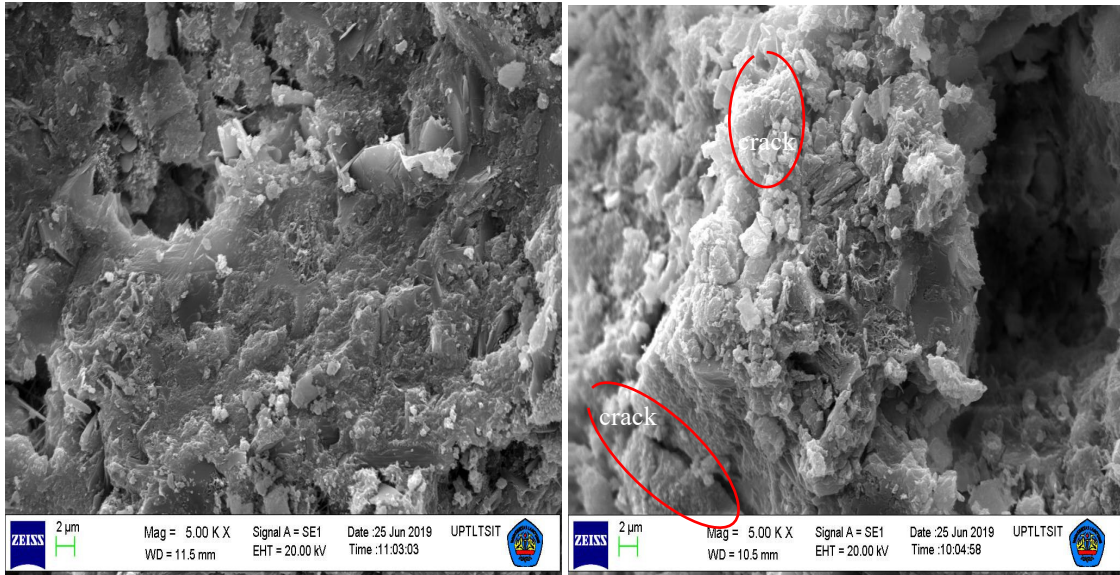
The dynamic viscosity of Ca, and Si in the submerged concrete in mud is 1-2 cps/eV smaller than the protected elements (Figure 5). This means for Ca and Si, the tangential force per unit area required to move one horizontal plane to another in the unprotected concrete is smaller than the protected condition. This indicates the solubility of Ca and Si and in Figure 6.4.b shows a stacked flat plane.

3.1.3 Scanning Electron Microscopy (SEM) Result

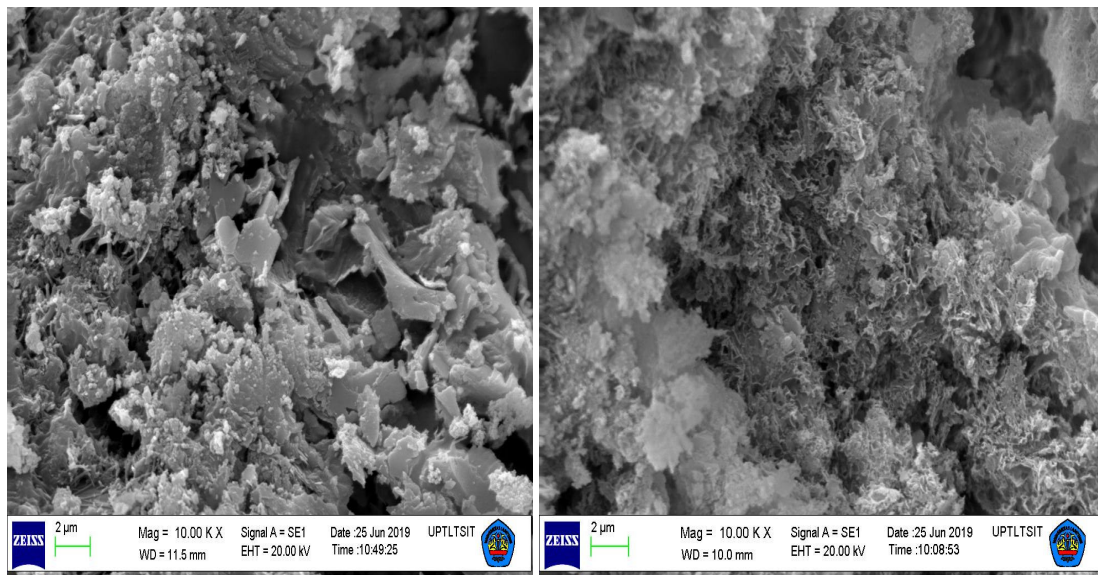
SEM for protected concrete and mud submerged concrete for magnitudes 2.00 K, 5.00 K, 10.00 K and 15.00 K are presented in Figures 6.1a, 6.2a, 6.3a, 6.4a and 6.1b, 6.2b, 6.3b, 6.4b, respectively.



(b) (b)
 Figure 6.1 SEM 2.00K: (a) Protected concrete, (b) Submerged in Mud



(a) (b)
Figure 6.2 SEM 5.00K: (a) Protected concrete, (b) Submerged in Mud



(a) (b)
Figure 6.3 SEM 10.00K: (a) Protected concrete, (b) Submerged in Mud

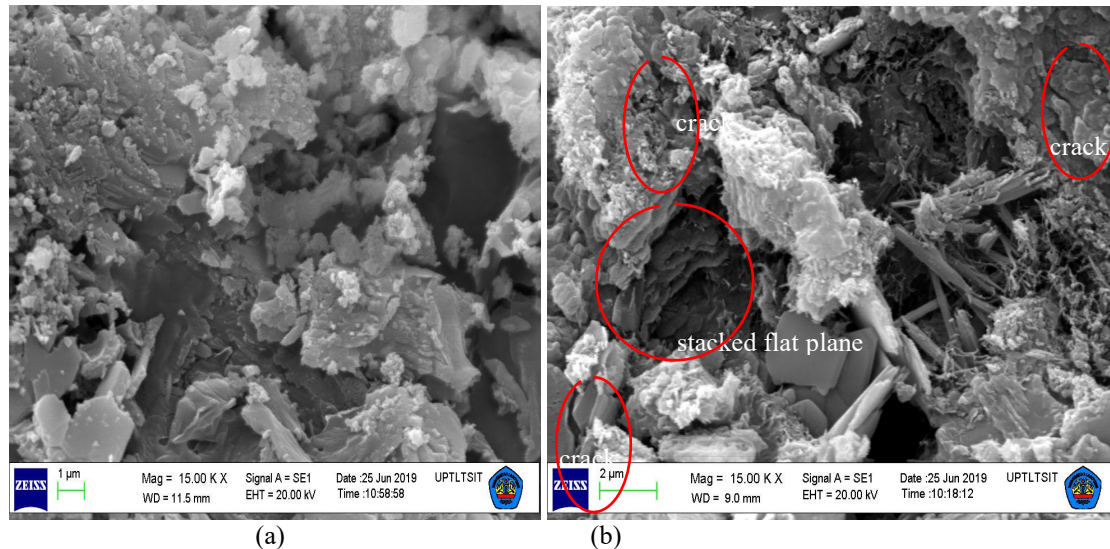


Figure 6.4 SEM 15.00K: (a) Protected concrete, (b) Submerged in Mud

The differences in the microstructure of protected and unprotected concrete are presented in Table 1.

Table 1. Differences in the microstructure of protected and unprotected concrete

Magnitude	SEM view of the concrete	
	Protected	Unprotected
2.00 K	Coarse & massive, small pores	The surface is rough, uneven, there are raised parts with small white lumps at the ends, many cracks, there are large pores and many small pores
5.00 K	CH crystals, clear CSH, very little calcite, deep visible pores	Blob with white tip, mass with many cavities, visible large cavity occupying $\pm 30\%$ visible area, large crack visible
10.00K	CH and CSH look very massive, with many pores and deep	White blobs occupy $\pm 25\%$, coarse mass with many small cavities, large cavities
15.00K	CH and CSH are very massive, with very little calcite, and deep pores	The white mass occupies $\pm 20\%$, the mass of the pile of flat plates, the mass is not still, large pores with a shape like a twig fracture, looks cracked.

3.2 Discussion

3.2.1 Infiltration of water containing clay

Under protected conditions, CH and CSH predominated a few white clumps were still visible (Figure 6.1a to 6.4a). SEM also shows the presence of ettringite (Figure 6.2a and 6.3a). The microstructure of the concrete submerged in mud is dominated by white lumps (Figure 6b). Upon closer inspection, it can be seen that the white lumps in the submerged concrete appear to be lumpier than the protected condition concrete, very little CH, CSH, and ettringite (Figure 6.1b to 6.4b). The agglomerates include Ca deposits (atomic mass 40% from the protected condition), Si (200% atomic mass from the protected condition), calcite (atomic mass C 127% from the protected condition), Al (atomic mass 148% from the protected condition) as shown in Figure 4. Friedel's salts, free bound chlorides, solid silica gel, CSH shards towards the surface, unstable Fe, atoms in the cell coordinate units, new minerals, metal hydroxides, silica shells, aluminates, sulfates, ettringite expands due to containing water, porosity 17-20% (Lagerblad 2001). All these compounds are under stress which causes the forces within them to repel.

Infiltration of water containing clay in this study came from one side. The water enters the pores of the concrete. At first, there was fast water infiltration, then the infiltration slowed down. Diffusion occurs through an almost stagnant liquid film, leaching occurs at the interface between the changed and unchanged concrete. The water itself contains carbonates and Mg to make the surface a rich silicate gel. As a result of this on the surface, CH dissolved and removed, a hollow shell after clinker appears (Figure 6.1b, 6.3b), a hydration reaction occurs from the un-hydrated cement present in the pore network. Calcium ions dissolve and enter the water to reduce the volume. The layer below the surface undergoes decalcification and densification. The predominant densification mechanism is through rearrangement of the solid particles "submerged" in and wetted by the low viscosity liquid, and through dissolution and re-precipitation of the solid (Yilmaz 2021). This makes mineral breakdown or cracks (Figure 6.1b, 6.3b, 6.4b).

3.2.2 Oxidation

The atomic mass O of the protected concrete was 51% of the sample submerged in mud (Figure 4). Clay contains SiO_2 , Al_2O_3 , Fe_2O_3 , K_2O , MgO , TiO_2 , Na_2O , CaO (Gonggo *et al.* 2001). The water that enters with the mud into the concrete is surface water. This water captures oxygen before it enters the concrete. Surface water contains more oxygen, carbon, bicarbonate, and carbon dioxide than deep water. Air also contains a lot of oxygen. This phenomenon because surface water contains a lot of oxygen. This reaction changes the pH of the solution. A decrease in pH causes the ettringite to dissolve and release sulfate (Lagerblad 2001).

This oxidation process causes Mg and Si ions to move freely so that it can affect the coordination of the atoms in the unit cell. In addition, the coordination of the atoms of the minor elements also affects the change in structure (Figure 6.1b, 6.2b, 6.3b, 6.4b). The minor elements include elements of Si, Fe, Al, and Ni. The oxidation process also causes the appearance of Fe^{3+} ions. Each Fe^{3+} ion pair is accompanied by a cation vacancy, resulting in coordination between the atoms of the unit cell. This unstable Fe atom causes 2 types of bonds to appear, namely 3 long Fe-O bonds and 3 short Fe-O bonds. In slurry submerged concrete, Fe appears in four atom energies with four types dynamic viscosity: 0.2 eV with 41 cps/ev, 0.6 eV with 41 cps/ev, 6.4 eV with 41 cps/ev, and 7.1 eV with 7cps/ev. There is no Fe in protected concrete. The Si, Ca, and O atoms in the slurry submerged concrete showed a dynamic viscosity of 1-2 eps/ev lower than the protected concrete, while the other atoms had the same value (Figure 5). This is because the muddy water that soaks the concrete contains oxygen (Figures 4 and 5). This shows that the microstructure of the concrete submerged in mud has really changed (Figure 6.1b, 6.2b, 6.3b, 6.4b).

3.2.3 Effect of pressure on calcium

The entry of water into the concrete causes a reaction of calcium hydroxide with other compounds, causing Ca in the cement paste and CSH to enter the water while the water contains mud in which Ca is present. Calcium also reacts with carbon and forms calcite. Calcite covers the entire surface of the cement paste and stabilizes the paste. The influx of Ca from CH into the water causes the portlandite to be depleted or decalcified. When the CH is depleted usually the porosity is 12-15% by volume and the porosity becomes continuous (Lagerblad 2001). This increase in porosity can be seen in Figure 6.1b, 6.2b, 6.3b, 6.4b.

Ca that enters the water requires 20mmol CaO to settle. Clay contains CaO, Si, and metal elements contained in clay such as Ca, Al, Fe, K, Mg, Na, thus triggering dissolved calcium to settle to form silica shells and metal hydroxides, in more quantities than concrete which is only submerged in water and conditions protected. Carbonate and Mg enter into the paste to bind Ca in the surface layer, Ca is adsorbed on the surface, forming Friedel's salt. Friedel's salt occupies a greater volume after crystallization in the pores of the concrete than the compound it replaces; fill pores; bind free chloride in the concrete, therefore; the free chloride present in the concrete was reduced. As long as the concrete still contains calcium hydroxide, it can still bind the chloride present in the concrete (Lagerblad 2001).

Silica and calcite shells are under pressure because they are submerged in water mixed with mud. Experimental studies show that calcium undergoes several opposite transitions under stress, namely from fcc with an interatomic distance of 3.94 angstroms, 4 atoms per unit cell, atomic packing factor of 0.74 to bcc with an interatomic distance of 5.3 angstroms, 2 atoms per unit cell and atomic packing factor 0.68 (Organov *et al.* 2010; Vlack 2001). Thus; the Ca atoms become less dense as well as the O atom. This makes the atomic mass of Ca submerged in mud only 40% of the protected sample and the dynamic viscosity distribution becomes shorter (Figure 4).

Under these conditions, the atom continues to be depressed. Pressure creates a repulsive force (Vlack 2001). The results of a single crystal X-ray diffraction experiment show Bragg reflections in various reciprocal spaces (Iizuka, 2013). Thus; the force due to the immersion pressure, the contraction force, the Ca draining force, the depositional force, and the formation of a new chemical element become repulsive forces. All these forces break bonds and appear as many cracks (Figure 6.1b, 6.2b, 6.4b).

Thus; what appears on the surface is precipitated calcium/portlandite, Friedel's salt, free chloride bound concrete, metal hydroxide, and silica shell (Lagerblad 2001). It appears as a white blob like Figure 6.1b, 6.2b, 6.3b, 6.4b. Qing *et al.* 2017 stated that white crystalline lumps on SEM photos in concrete containing fly ash were gypsum and mirabilite ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$).

3.2.4 Silica

Oxidation makes the microelements Si, Al, Fe, and Ni move freely and affect the coordination of atoms. The increase in the mobility of Si and Al causes the porosity to also increase. The movement of Si due to oxidation and the depletion of CH, the depletion of Ca from CSH, and the incorporation of CH and CSH with other compounds makes CSH unbalanced to be incomplete and destroyed. This makes the porosity increase rapidly, can reach 17-20%, and become continuous. The concrete becomes less dense, the pore diameter becomes large (Figure 6.1b, 6.2b, 6.3b, 6.4b).

This happens until the Ca concentration is very low. Thus CaO/SiO_2 decreases, making the CSH structure change. If the decrease in CaO/SiO_2 is close to 1, CSH remains physically intact, but if Ca continues to be

depleted and, CSH breaks down and moves to the surface. CSH collapses to form a dense silica gel layer, shrinks, the porosity increases markedly, Si becomes easily soluble. The Si atomic mass of the mud submerged concrete is twice that of the protected concrete because the mud also contains Si (Figure 4).

The dissolved Ca causes the local pore water pH to change. pH less than 10.5 makes ettringite disappear releasing sulfate ions. The sulfate moves inward to form new ettringite, forming the aluminate phase, the ettringite expands because it contains water (similar to sulfate attack). The structural changes of the cement paste will affect the porosity, which in turn affects the diffusivity (Lagerblad 2001). Thus; on the surface, there is a dense layer of silica gel.

3.2.5 Carbon

Carbonate appears with the same dynamic viscosity between protected and submerged in mud concrete but with an atomic mass of C in the mud submerged condition 1.27 times greater than the protected condition (Figures 4 and 5). This is caused by the entry of Carbon from the clay into the sample submerged in the mud. The structural changes of the cement paste will affect the porosity, which in turn affects the diffusivity (Lagerblad 2001). Diffusion of Mg ions leads to enrichment of Mg- in silicate gels. Protected concrete and submerged in mud does not contain Mg (Figures 4 and 5). Water with Mg that enters the alteration zone can remove Mg. The absence of Mg falls in zones 4,5 and 6 (Lagerblad 2001).

3.2.6 Microprestress in concrete submerged in muddy water

There is product hydration growth, capillary stress, disjoining pressure, and surface tension or microprestresses in normal concrete. Because it has been submerged for more than 10 years, the hydration process is considered to have ended, meaning that the hydration product growth force is considered non-existent. In concrete that is submerged in water mixed with mud for years, the pores of the concrete are filled, this does not allow the liquid to rise or fall, thus the capillary attraction force can be neglected. In the presence of water in the concrete, there is a repulsion to the van der Waals attraction. The decrease in pH causes the thickness of the film to increase, causing a decrease in the disjoining pressure. The water in the pores of the concrete is filled and the water between the hydration products is also ever-changing due to the depletion of Ca. So; the water that is at rest also decreases, so the surface tension is also getting smaller. Thus; the microprestresses force is ignored.

4. Conclusion

The compressive strength of concrete submerged in mud for 10 years is only half that of protected concrete. The microstructure of the mud submerged concrete showed significantly less CH and CSH than the protected concrete and was dominated by white lumps which looked more brittle and the pore diameter looked larger than the protected concrete. The immersion of the mud makes the atomic masses of Ca and Au decrease but the atomic masses of O, Si, C, Al increase, and Fe appears.

The strength and durability of concrete are affected by porosity, permeability, pore size, and distribution, therefore; the microstructure of concrete (porous structure of cement paste, and the interface zone between cement paste and aggregate) has an important role in determining the performance and durability of concrete.

The degradation is mainly caused by the oxidation of surface water, the depletion of Ca from portlandite which in turn depletes Ca from CSH. Ca depletion in slurry submerged concrete occurs under pressure. Due to pressure, the atomic density of Ca changes from 4 to 2 per unit cell with atoms 1.3 times farther apart. This results in the atomic mass remaining 40%. The depleted CH, followed by CSH collapse increases the porosity. The presence of Al in the clay adds to the atomic mass and porosity. This makes the capillary pores enlarge, and makes them continuous. The clay makes the silica shell have an atomic mass of Si two times the protected condition, the contraction force is greater, and the repulsion force due to compression is greater. The unstable Fe causes structural changes in the concrete. This condition makes the compressive strength of the concrete submerged in mud only 50% in a protected condition.

The right way to prevent concrete from degrading its strength is to make the concrete a minimum of water permeable and protect it with a waterproof layer.

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AUTHORS' NOTE

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