Effect of citric acid on setting-time and compressive strength of concrete

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Abstract. Citric acid is an organic acid naturally present in fruits, one of the ingredients that can be used as a retarder in a concrete mixture. As retarder serves to slow the timing of concrete hardening. In the implication the use of this material affects the time of hardening and compressive strength, therefore need a better control to get the optimal value. The test sample used was 36 with variation of addition of citric acid 0%, 0.15%, 0.3%, 0.45% for the weight of cement. The results obtained the greater the addition of citric acid the longer the hardening of concrete and the greater workability of concrete. The maximum value of compressive strength of concrete is in the addition of 0.15% citric acid with an increase in compressive strength of 82.2% of the nominal concrete.

1. Introduction

Chemical Retarder Admixture brand is an additional chemical that serves to slow down the initial cement setting time so that the mixture will still be workable for a longer time. The way a retarder works is by wrapping the cement grains with OH thus slowing the initial reaction of the hydration. The formation of Ca salts in water reduces Ca concentration and slows crystallization during the hydration phase [1].

The additives that can be used as retarders include sugar, sucrose, sodium gluconate, glucose, citric acid and tartaric acid [1]. The addition of sugar to the concrete can extend the optimum initial setting time of the concrete. In the variation of 0% to 0.45%, the optimum length of the initial setting time continues to increase [1]. The addition of sugar levels can longer the time of hardening of the cement to the level of 0.15%, after which the hardening time again decreases when more sugar is given [2].

Portland / calcium aluminate blended cement (PC / CAC) was combined with citric acid or lactic acid as additives to the antioxidant effects of PC / CAC as a potential fast hardening and low cost repair material for concrete [3]. Mortar specimens with the carboxylic acid additives of either 0.5%, 1% or 3% by weight, prepared with a binder: sand: water ratio (by weight) of 1: 3: 0.5 experiments analyzed revealed Portlandite crystals were the major hydrate phase in PC / CAC with lactic and citric acids.

Lactic acid below 2% wt. improved both compressive and flexural strength at early ages due to improved crystallinity of the calcium hydroxide crystals. Combined with its inherent rapid setting
time, PC / CAC blended cements have a potential to be developed into a suitable repair material for concrete.

The Citric acid effectiveness depends strongly on the amount added to the interaction with other raw materials in particular, in particular calcium aluminate cement [4]. Citric acid partially inhibits the hydration binder from hemihydrate to dihydrate, as observed by X-ray diffraction analysis [5].

Sugar content can be used as a retarder has been researched by researchers. Citric acid is an organic acid that is naturally found in fruits such as oranges, pineapple, and pear[6], [7], [8], [9]. Citric acid is first extracted and crystallized from citrus fruits, so extracted citric acid from these fruits is known as natural citric acid. Citric Acid has the chemical formula C$_6$H$_8$O$_7$ or CH$_2$(COOH) • COH (COOH) • CH$_3$(COOH) [10].

Citric acid is an organic acid that is widely used in the food, beverage, pharmaceutical, cosmetic, agricultural and chemical industries. Citric acid can be produced through the fermentation process of citric acid-producing microorganisms. Aspergillus niger is a microorganism that can be used in the production of citric acid. The use of citric acid as a retarder is an environmentally friendly material.

Environmentally friendly alternative materials that could substitute cement both partially or totally have been developed using waste material such as fly ash [11]. According to the Building Constructive Materials and Civil Engineering Guidelines Number 22 / SE / M / 2015 concerning guidelines for the use of chemical admixture in concrete, the added ingredients are divided into several categories according to their chemical composition, including: 1) lignosulphonic acids and other salts for example Na, Ca, or NH$_4$, 2) Hydro-carboxylic acids and their salts, 3) Carbohydrates - carbohydrates and sugars, 4) Inorganic salts based on flourates, phosphates, oxides, borax, and magnesium salts [12].

The purpose of this study was to determine the optimal value of adding citric acid to concrete with cement bonding time, sloughness and concrete compressive strength.

2. Theoretical Underpinnings

2.1. Cement Setting Time (Vicat Test)
The time of cement bond is the time it takes for cement to react with water. The time of cement bonding is divided into two, namely the initial setting time and the final setting time. The definition of initial setting time in accordance with Indonesian Standart is the time required by cement paste to change its properties from liquid to solid conditions. While the final setting time of cement is the time when the penetration of the vicat needle is not visually visible [13]. The initial setting time can be seen when the vicat needle gets 25 millimeters of penetration while the final setting time is when the vicat needle cannot penetrate [14].

2.2. Slump Test
Slump test is an empirical test used to determine consistency / stiffness from a mixture of fresh concrete to determine the level of workability. Stiffness in a concrete mixture shows how much water is used. For this reason the slump test shows whether the concrete mixture is less, more, or enough water. In a mixture of concrete, the water content is very considered because it determines the level of workability or not. Concrete mixtures that are too liquid will cause the concrete quality to be low and long dry time. While the concrete mixture that is too dry causes the mixture to be uneven and difficult to print [15].

2.3. Compressive Strength of Concrete
The compressive strength of concrete will increase with increasing age of concrete. The strength of the concrete will increase quickly up to 28 days, but after that the increase will be law. The compressive strength of concrete in certain cases will continue to increase for several years upfront. Usually the compressive strength of the concrete plan is calculated at 28 days. Concrete compressive strength identifies the quality of a structure. The higher the level of the desired structural strength, the higher the quality of the concrete produced. Nawy [24] argues that the parameters that most influence the strength of concrete are: Quality of cement, proportion of cement to mixture, strength and cleanliness of aggregates, interaction or adhesion between cement paste and
aggregate, sufficient mixing of materials concrete forming, correct placement, solution, concrete compaction, and concrete treatment.

3. Experimental

3.1. Materials

The materials used in this research were cement, sand, gravel, and citric acid. The cement used had to meet prescribed requirements in the specification of group A construction material. The cement used was Gresik cement. The sand used was taken from River Kaliworo. It met the prescribed requirements in the specification of group A construction material. [16]. The coarse aggregate used was taken from PT Pancadarma Puspawira, it met the prescribed requirements in Indonesian Standart [17], [18]. Retarder or added material used in this research is Elephant brand citric acid produced by PT Budi Starch & Sweetener Tbk.

3.2. Experimental Procedure

3.2.1. Examination of Material

The sand examination was done through the following steps: the examination of water level, specific gravity, unit weight, organic content, mud content, and sand gradation which had to meet the prescribed requirements of Indonesia standard. The gravel examination was done through the following steps: the examination of specific gravity, abration, and gravel gradation which had to meet the prescribed requirements of Indonesia standard.

3.2.2. Mix design concrete

At the calculation stage of the concrete mix planning with or design of a mixture of concrete adding citric acid as a retarder with variations in addition of 0%, 0.15%, 0.30%, 0.45% of the weight of cement The concrete mix plan calculation is done to determine the need for gravel, sand, cement and water. The mixed method used in this study is to use the Indonesian standart mix design method [19].

3.2.3. Setting Time Test

Testing step of the initial cement setting time using vicat test apparature. This test is carried out using American standard. [20] The following is Vicat Apperature or Vicat Tool which is used to know the time of binding of cement:

![Figure 1. Vicat Tool (1 mm diameter needle)](image-url)
3.2.4. Workability Test
Testing is done using Abrams cone and steel rod as crusher, testing is conducted to know the value of slump concrete (workability) based on Indonesian Standard [6].

![Abrams Cone Diagram]

**Figures 2. Abrams Cone**

3.2.5. The Compressive Strength Test of Concrete
Concerning the compressive strength test of concrete. Compressive strength of concrete bricks is the amount of load per area unit which cause the specimen to break if it is loaded with certain amount of compressive force produced by compressing machine. The compressive strength test of concrete was conducted 28 days after the production of the specimens, pursuant to the prescribed requirements of Standar Nasional Indonesia by using concrete compressive test apparatus.

The compressive strength was calculated by using equation as follows:
\[ \sigma = \frac{P}{A} \]

where \( \sigma \) = compressive strength, \( P \) = load, \( A \) = area of specimen

4. Results and Discussion
The result of sand material test is shown in Table 1. Based on the examination result, the sand already met the requirements to be used as construction material. One of the requirements of sand, which is good to be used as construction material is having a specific gravity of 2.4-2.9 [6].

| Properties       | Values  |
|------------------|---------|
| Water Content    | 6.67%   |
| Specific Gravity | 2.5     |
| Fineness Modulus | 3.69    |
| Zone             | 2       |
| Absorption       | 1.8 %   |

The result of gravel material test is shown in Table 2. Based on the examination result, the gravel already met the requirements to be used as construction material. One of the requirements of gravel, which is good to be used as construction material is having a specific gravity of 2.4-2.9 [10].

| Properties       | Values  |
|------------------|---------|
| Abration         | 5.206 % |
| Specific Gravity | 2.53    |
| Fineness Modulus | 4.905   |
| Zone             | 2       |
| Absorption       | 1.493%  |
The cement testing carried out in the study included normal consistency testing and initial cement binding test. Normal consistency testing is done to determine the water content needed to react to each unit of cement needs. [21] The method of obtaining a normal consistency value is to do a minimum of 5 trials and with the same results, so that in this study a normal consistency value of 27.33% was obtained. The initial setting time test is conducted to determine the time needed for cement to react with water to be plastic. The data of the results of initial setting time test is shown in Figure 3.

![Figure 3. Result of Setting Time](image)

Based on the results of data analysis in testing the first hypothesis it was concluded that there was an effect of adding citric acid as a retarder with a percentage of the weight of cement 0.00%; 0.15%; 0.30%; 0.45% of the initial setting time of cement. This conclusion is in accordance with the results of the research that has been conducted. It can be defined that with more and more levels of addition of citric acid as a retarder in cement, the length of time the initial setting of cement is produced. Slowing the setting time on cement occurs because the citric acid reacts with water and then the water reacts with cement so that a different hydration process occurs according to the variation of addition or level given. In simple terms the reaction that the retarder will wrap the cement grain with OH- so that it causes a slowdown in the initial reaction of the hydration of cement. Another factor that influences the time of cement bonding or setting time other than the ambient temperature is the water content or FAS (cement water factor) used to react a number of cement [22].

The hydration process of cement in fresh concrete requires approximately 25% of the weight of cement [23]. To determine the amount of water or water content needed in the setting time test (vicat test), normal consistency testing is carried out. In this study using ASTM C-187 standard so that the required water content is 82 milliliters or 27.33% of the weight of cement. Whereas for the need for citric acid as an added ingredient is to calculate the percentage percentage of cement content multiplied by the weight of the cement to be tested at time (according to ASTM).

Examination of fresh concrete slits is done by testing the slump using the Abrams cone. Data of the slump test is shown in Figure 4.
Based on the results showed that there was an increase in concrete slope which was directly proportional to the addition of citric acid. Workability can be interpreted as concrete thickness. The thicker the fresh concrete, the more difficult the workability level. [24] The elements that influence workability are the amount of mixing water, cement content, gradation of sand and gravel mixtures, coarse aggregate granular forms, and compaction methods or methods. The difference in slump values that occur in this study can be caused by the type of added material and the percentage of added material given. This is consistent with the results of research the addition of certain levels of sugarcane drops resulted in an increase in slump value. [23] On the addition of sugarcane drops with an addition of 0%, 0.2%, 0.8%, respectively, the slump values increased by 7.5 cm, 10.8 cm and 13.4 cm.

The result of compression strength test of concrete is shown in Figure 5.
that the compressive strength value after the variation of 0.15% has decreased, it happened because of some influencing factors. According to Nurlita Pertiwi the factors that influence the strength or quality of concrete are the cement water factor or the ratio of water and cement weight, aggregate type and gradation, cement quality and concrete curing. In addition, as one of the other factors that influence the compressive strength of concrete is added material (admixture) and the level or percentage of material use added. Improper addition of retarding admixture can result in the nature of the concrete to be ugly or not as expected. Based on the graph figure 3-5 test results the average compressive strength of concrete aged 28 days produced different values. This happens because the percentage of addition of citric acid as a different retarder. The results of the 28 days age concrete compressive strength test can be proven by looking at the results of the setting time testing and slump testing because they are interrelated. In principle, the longer the initial setting time of cement occurs, the higher the slump value. However, a high slump value is not necessarily followed by a high compressive strength value. This happened because the percentage of retarder admixture was added improperly so that in the reaction process cement was unable to hydrate and bind aggregate well because it was wrapped by too many retarders. This inability results in aggregates not being distributed well and also resulting in a squeeze or separation between aggregates of fresh concrete dough. The result of the failed reaction process is that after dry the concrete has a low compressive strength.

5. Conclusion
The results obtained the greater the addition of citrit acid the longer the hardening of concrete and the greater concrete (workability) of concrete. The maximum value of compressive strength of concrete is in the addition of 0.15% citrit acid with an increase in compressive strength of 82.2% of the nominal concrete.

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