Formulation and Evaluation of Effervescent Granules Ethanol Extract of Andaliman Fruit (*Zanthoxylum acanthopodium* DC) with Combination of Citric Acid-Tartaric Acid and Sodium Bicarbonate

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**ABSTRACT**

The combination of citric acid-tartaric acid and sodium bicarbonate in effervescent granules has an important role in providing an effervescent effect. Citric acid and tartaric acid are organic acids that can react with sodium bicarbonate when they come in contact with water. This reaction results in the release of carbon dioxide gas, which has a splitting effect and makes the effervescent granules dissolve rapidly in water. Research on the formulation and evaluation of effervescent granules of andaliman fruit ethanol extract with a combination of citric acid-tartaric acid and sodium bicarbonate aims to optimize the stability, solubility, and release of the active ingredients from the granules. This study is an experimental research. Evaluation of effervescent granules is carried out in the form of an organoleptic test. This test can be observed directly from the granules through the shape, color, aroma, and taste produced. Granular specific gravity test, namely real specific gravity, compressed specific gravity, flow rate test, angle of repose test, and compressibility. Tests for dispersion time and pH tests were carried out in this study. FI, FII, and FIII meet the requirements of the organoleptic test, compressibility, flow rate, angle of repose, dispersion time, and pH test. Even though the pH test for the 3 formulas found a pH range of 5.5 to 5.7, but still declared to meet the requirements because it is still close to neutral pH. The best influence on the physical properties of granules effervescent, namely in FIII, as seen in the organoleptic test, flow rate, angle of repose, and dispersion time.

**1. Introduction**

Effervescent granules are a pharmaceutical dosage form that has its own uniqueness. These preparations contain a combination of active ingredients with effervescent agents, which can provide a splitting effect and release gases when the granules are diluted in water. In this case, effervescent granules developed using the ethanol extract of andaliman fruit (*Zanthoxylum acanthopodium* DC) with a combination of citric acid-tartaric acid and sodium bicarbonate are an attractive option in the development of herbal preparations that can optimize the efficacy and stability of the active ingredients. Andaliman fruit is a plant that grows in Indonesia, especially in North Sumatra. This fruit contains bioactive compounds such as alkaloids, flavonoids, and essential oils, which have been shown to have various pharmacological benefits, including anti-inflammatory, antimicrobial, and antioxidant properties. The use of ethanol extract from andaliman fruit in effervescent granules is expected to provide greater health benefits for users."
react with sodium bicarbonate when they come in contact with water. This reaction results in the release of carbon dioxide gas, which has a splitting effect and makes the effervescent granules dissolve rapidly in water. By having an effervescent effect, effervescent granules can increase the solubility and bioavailability of active ingredients, thereby accelerating their absorption and therapeutic effect.  

Research on the formulation and evaluation of effervescent granules of ethanol extract of andaliman fruit with a combination of citric acid-tartaric acid and sodium bicarbonate aims to optimize the stability, solubility, and release of the active ingredients from the granules. The evaluation was carried out through physicochemical parameters such as particle size, release rate, solubility, and physical and chemical stability. Thus, this research is expected to provide useful information in the development of effective and stable andaliman fruit ethanol extract-based effervescent granule preparations.

2. Methods

This study is an experimental research. This research was conducted at the laboratory of the Faculty of Pharmacy, Universitas Sumatera Utara. Andaliman fruit was obtained from Simalungun Regency, North Sumatra, Indonesia, and plant determination was carried out at the Universitas Sumatera Utara. Andaliman fruit is washed and dried and used as simplicia powder. The simplicia powder was macerated with solvent ethanol 70% for 3x24 hours. Macerate was carried out by an evaporation process with a rotary evaporator to obtain a thick extract of andaliman fruit. The effervescent granules were prepared in three formulations: Formula I (F1) consisted of 2.5% andaliman extract, 9.5% citric acid, 19% tartaric acid, 32.3% sodium bicarbonate, 3% PVP, 0.5% aerosil and added lactose to 100 percent. Formula II (FII) consists of 2.5% andaliman extract, 10% citric acid, 20% tartaric acid, 34% sodium bicarbonate, 3% PVP, 0.5% aerosil, and added lactose up to 100 percent. Formula III (FIII) consists of 2.5% andaliman extract, 10.5% citric acid, 21% tartaric acid, 35.7% sodium bicarbonate, 3% PVP, 0.5% aerosil, and added lactose up to 100 percent. Evaluation of effervescent granules is carried out in the form of an organoleptic test. This test can be observed directly from the granules through the shape, color, aroma, and taste produced. Granular specific gravity test, namely real specific gravity, compressed specific gravity, flow rate test, angle of repose test, and compressibility. Tests for dispersion time and pH tests were carried out in this study.

3. Results and Discussion

Organoleptic test

Granules that have been processed have similar characteristics starting from their color, shape, and aroma. The shapes produced by them have the same characteristics, namely in the form of granules. This is obtained from a mesh sieve that has the same size as the three formulas. The colors produced from the three formulas are all yellowish-green. This yellowish-green color is obtained from the andaliman extract, which was originally blackish-green when mixed with other ingredients to produce the yellowish-green color. As for the smell, the three formulas also have a similar aroma, namely the distinctive aroma of andaliman. The distinctive smell itself comes from the andaliman fruit.

But for taste, the three formulas have different tastes. The formula I itself has a distinctive taste from andaliman. Formula II has a more sour and slightly sweet taste due to the greater concentration of citric acid and tartaric acid used than formula I, and formula II still has the distinctive taste of andaliman itself. While formula III has the sourest taste of the three because the concentration of citric acid and tartaric acid used is higher than in the two previous formulas. This formula also still has a distinctive taste from its mainstay.

Granule-specific weight test

The compressibility test value of formula I = 20.93%, formula II = 23.25%, and formula III = 23.25%. Formula I show quite good compressibility
results because it has yields ranging from 16% to 20%. For formulas II and III, the compressibility results are quite good because they are located in the range of 21% to 25%. Of the three formulations, the formula I had the best effect when compared to formula II and formula III on the granule compressibility test because the use of the two acids increased the shape of the granules and compacted the granules.

Granule shape can affect good compressibility. Particle shape, particle size, and particle size distribution play a role in determining the size of the compressibility index. The small value of the compressive index shows that the granule has a good ability to organize itself. When compressed, there is no significant volume shrinkage. In this compression test, the capacity of the powder to arrange itself and occupy the empty space between the particles is greatly influenced by the porosity, density, and particle size.\(^\text{11}\)

**Flow rate test**

Flow rate tests on formulas I, II, and III obtained the following results: formula I had a flow time of 7.44 seconds, formula II had a flow time of 6.64 seconds, and formula III had a flow time of 6.34 seconds. All of these results met the requirements of the granule flow rate test, i.e., flow time <10 seconds. Therefore, granules are effervescent. The products produced from the three formulas are considered good in their flow properties. Of the three formulas above, formula III has a flow rate that is superior compared to formula I and formula II. This situation is caused by the use of greater tartaric acid. Granule flow rate is affected by various factors, including shape, size, porosity, density, frictional force, and experimental conditions. These factors affect the ability of the granules to flow. Different molecular weights and densities can affect the flow of granules, whereas granules with lower molecular weights and densities tend to flow more easily. Comparison of acid and base levels in the three different formulations as good influence granule flow rate. Tartaric acid has a higher density than citric acid, so granules containing tartaric acid tend to have a higher density. As a result, the granule flow rate in the three formulas is different. Differences in the ratio of acid and base content in the formulation also cause variations in the flow rate of the granules.\(^\text{14-16}\)

**The angle of repose test**

The angle of repose test on formula I; 25.73°, formula II; 25.46°, formula III; 25.07°. This means that if all the requirements for each formula have been met, namely <30°, it means that it can flow freely. Of the three formulas above, formula III has the best effect on formula I and formula II. The smaller the angle of repose, the better the flow properties of the granules. The result of formula III granule flow time was the fastest, namely 6.34 seconds. The size and number of granules, the diameter of the funnel, the method of pouring, and the influence of vibration affect the size of the angle of repose formed. In addition, the angle of repose formed is also affected by the use of citric acid and tartaric acid due to the greater cohesive forces between the particles.\(^\text{17,18}\)

**Dispersion time**

Dispersion time in formula I; 4.8 minutes, formula II; 2.35 minutes, formula III; 2.23 minutes. This means that if the 3 formulas have passed the requirements for the dispersion time test, namely <5 minutes, it means that they are perfectly dispersed. Of the three formulas above, formula III has the fastest dissolving speed than formula I and formula II. This is because of the use of more acid-base concentrations than in formula I and formula II. The attractive forces between the solute particles and the solvent particles greatly affect the solubility. In this process, there is a withdrawal of dissolved molecules by dissolved molecules to move away from each other. So it will happen until. Finally, the condition of the solvent molecules can no longer shrink the solute molecules (saturated conditions), and there is a precipitate. The composition and conditions during the drying process, the temperature of the solvent, and the method of mixing also affect the solubility of the powder. Dissolving speed is also affected by the use of citric
acid. In cold water, citric acid dissolves faster than in warm water. Granule porosity is also related to dispersion time. The large porosity of the granules will enlarge the voids between the particles so that the liquid can immediately enter the granule structure and pressure the granules to melt.19

**pH test**

pH test results on formula I; 5.7, formula II; 5.6, formula III; 5.5. These results indicate that the three formulas above are classified as acidic because of the requirements of the pH test. Namely, if the pH measurement results are close to 6-7 (near neutral), it is said to be good. But the results of the three formulas above are not too acidic and are still said to be good because they are close to a neutral pH, so they can still be said to meet the requirements. Of the three formulas above, the formula I have the best pH value because it is closer to a neutral pH than formula II and formula III. This is due to the use of a smaller acid concentration than formula II and formula III.

CO₂ formed during the reaction effervescent in water half will dissolve and become carbonic acid, then H⁺ ions will decrease. This makes the solution acidic and causes the pH value to decrease. In granules, effervescent contains acids and bases. When each of these components reacts with water, then in solution, the two compounds will be released. The low pH of the solution will cause a sour taste. Weak base compounds that are separated from sodium bicarbonate can be seen from the presence of air bubbles coming out due to the reaction between the acidic and basic elements.20

**4. Conclusion**

FI, FII, and FIII meet the requirements of the organoleptic test, compressibility, flow rate, angle of repose, dispersion time, and pH test. Even though the pH test for the 3 formulas found a pH range of 5.5 to 5.7, but still declared to meet the requirements because it is still close to neutral pH. The best influence on the physical properties of granules effervescent, namely in FIII, as seen in the organoleptic test, flow rate, angle of repose, and dispersion time.

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