VARIATION CONCENTRATION OF CITRIC ACID AS ACID SOURCES ON THE PHYSICAL PROPERTIES OF THE PERICARP MANGOSTEEN (*Garcinia mangostana* L) EXTRACTS EFFERVESCENT TABLET

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ABSTRACT

Aim: Citric acid is an acid source in effervescent tablet which reacts rapidly with solvent. It can increase dissolving time. In this research, extract was made into effervescent tablet. The research aimed at knowing increased concentration of citric acid as acid source can improve the physical characteristic of effervescent tablet.

Methods: Pericarp mangosteen macerated by water as solvent, and the extract was made into powder by spray dryer method. Dry extract was made into 5 formulas effervescent tablet with the different concentrations of citric acid, 15%, 20%, 25%, 30% and 35% as acid sources. The evaluation of tablets quality included organoleptic evaluation, weight uniformity, size uniformity, hardness, friability, and dissolving times.

Results: The result of dissolving time was subquently 10.50±0.57, 4.52±1.04, 3.32±0.13, 3.13±0.14, and 3.42±0.14 minutes. The value of dissolving time between the concentration of citric acid were significanly different (ANOVA, $p<0,05$).

Conclusion: It can be concluded that the level of concentration of citric acid affected on dissolving time of the pericarp mangosteen (*Garcinia mangostana* L.) dry extract effervescent tablets.

Keywords: Mangosteen, Effervescent tablet, Citric acid

INTRODUCTION

Mangosteen (*Garcinia mangostana* L) contains xanthon which is useful for body health. Xanthon is proven to have very high antioxidant activity that exceeds the strength of vitamin C and vitamin E. There are two types of xanthon in the mangosteen pericarp which are most beneficial to health are alpha mangostine and gamma mangostine [1]. Mangosteen pericarp is very potential to be processed into health products. One alternative to processed mangosteen products is an effervescent tablet dosage form. Effervescent tablets will quickly dissolve in water, produce clear solutions, and give a sparkle effect or like the taste of soft drinks so that the effervescent dosageforms are much preferred by public.

The source of acid used is citric acid and the base source used is sodium bicarbonate. Citric acid is the most common food acid used in effervescent preparations because it is easy to

obtain, abundant, relatively inexpensive, very soluble, has high acid strength [2], has an electronegativity greater than tartaric acid so it is easy to release hydrogen ions which will quickly react with bases. The easier it is to release hydrogen ions, the faster it reacts, increasing the time to dissolve effervescent tablets.

In previous research on the use of citric acid, as an acid source it could affect the physical properties of effervescent granules, increasing the time to dissolve from dried mangosteen pericarp compared to the use of tartaric acid or a combination of citric acid and tartaric acid [3]. In this study the concentration of citric acid used in each formula was 15%, 20%, 25%, 30% and 35%.

This study aims to determine whether the dried extract of mangosteen pericarp can be formulated into effervescent tablet preparations that meet the requirements and see the variation of citric acid concentration as an acid source for the physical properties of effervescent tablets including hardness, dissolving time, and organoleptic such as shape, smell, and taste. The success of this research is expected to be the development of the utilization of mangosteen pericarp on other pharmaceutical dosageform and accepted by public.

MATERIALS AND METHODS

Materials

Mangosteen pericarp extract, maltodextrin, polyvinylpyrrolidone (PVP), aspartame, lactose, citric acid, 96% ethanol, sodium bicarbonate, polyethylene glycol 6000, and aquadest.

Evaluation of Mangosteen Pericarp Extract

1. Organoleptic test: test of color, odor and taste
2. Flavonoids test [4].
3. Loss on drying (LOD) test [4].
4. Ash residue test [4].

Formulations of Effervescent tablets

The tablets made in 5 formulas as shown on Table 1.

Tabel 1: Formula of Mangosteen Extract Effervescent Tablet

Material	Function	F 1	F II	F III	F IV	FV
Dry Powder Extract	Active ingredient	16	16	16	16	16
Citric Acid	Acid Source	15	20	25	30	35
Sodium Bicarbonate	Base Source	20	24	30	36	42
PVP	Binder	1	1	1	1	1
Aspartame	Sweetening agent	1	1	1	1	1
PEG 6000	Lubricant	3	3	3	3	3
Lactose	Filler	ad 100	ad 100	ad 100	ad 100	ad 100

Tableting Process

Effervescent tablets were made in special conditions with 25% relative humidity at 20-25°C [2]. All ingredients were dried previously in the oven for 1 hour, then weighed as much as in formula ordered.

Acid Components: Dry extract powder mixed with citric acid, aspartame, some lactose and some PVP, added slowly 96% ethanol until the mass of the tablet can be shifted into granules. Then sieved with number 14 sieve and dried in an oven at 50°C for ± 18 hours [5]. The dried granule was sieved with number 16 sieve.

Base Components: Sodium bicarbonate was mixed with the remaining lactose and PVP residues, added slowly 96% ethanol until the mass of the tablet can be sieved into granules. The next step was the same as in the acid component.

Acid components and base components were mixed until homogeneous. The mixed granule added PEG 6000 in the container. The results of the mixed granules obtained were evaluated and made into tablet with the weight of each tablet 5 g. The tablets obtained ready to evaluate.

Evaluation of Granules

Flow time: Granules weighed as much as 100.0 g and then put in a funnel. Calculate the time needed for the granule to flow [2].

Angle of repose: Performed when testing the flow time by measuring the height and diameter of the cone of the granule bed. To calculate it, the formula number (1) is used [6].

$$\tan \alpha = h / r \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

Compressibility test: The granule was inserted into a 100 ml measuring cylinder. Then beat 500 times, observed volume before and after being hit [2].

Particle size distribution: Weighed as much as 100 g of granules then put into a multilevel sieve. Sift at a frequency of 30 Hz for 25 minutes, then the weight of the granules left in each sieve was weighed [6].

Loss on drying: Took 1.0 g of granule in a wide-mouthed container, put it in the moisture balance, turned on the appliance, then observed the percentage loss in weight [4].

Tablet Evaluations

Size uniformity: Measured the diameter of the tablet and then measured the thickness of the tablet for 20 tablets [7].

Weight uniformity: A total of 20 tablets were weighed, then weighed back one by one. The average weight of the tablet was calculated. Calculated the percentage difference between each tablet to the average weight. Then match the requirements as requested in the Indonesian Pharmacopoeia [8].

Tablet hardness: A total of 10 tablets were measured using a digital hardness tester, the results were observed [2].

Friability of tablets: A total of 20 tablets that have been cleaned from dust are weighed. The tablet is then inserted into the friability tester and then rotated for 4 minutes. After that the tablet was cleaned from dust again and weighed. Calculated differences in weight before and after treatment [7].

Dissolved time: A tablet was put into 200 mL of water. Observe the time needed until the tablet dissolves [9].

Data Analyses

The dissolved time data of the tablet was analyzed with one-way ANAVA statistics. Followed with the Tukey test with a 95% confidence level (α= 0.05) to find out the significant differences between formulas.

RESULTS AND DISCUSSION

Results of Extract Evaluations

Evaluation of dried extracts of mangosteen rind, obtained from PT. Phytochemindo Reksa, Bogor, West Java, includes organoleptic tests, evaluations of flavonoids, LOD and ash residue. The results of the characterization of dried mangosteen pericarp extract can be seen in Table 2. The results of granule evaluation can be seen in Table 3 and the results of evaluation of the tablet can be seen in Table 4.

Table 2: The Results of Characterization of Dry Mangosteen Pericarp Extract

Observation	Results
Form	Powder
Color	Yellow-Brownies
Smell	Specific
Taste	Specific
LOD	1.5 %
Identification of flavonoid	positif
Ash Residue	0.69%

Table 3. The results of Granule Evaluations

Evaluations	F1	F2	F3	F4	F5
Flow Time (Second)	4,54	5,82	5,84	7,29	6,39
Angle of Repose (º)	28,06	28,09	28,36	30,39	29,22
Compressibility (%)	5,66	4,66	4,66	3,99	4,6
LOD (%)	3,23	1,65	0,6	0,61	0,97

Results of Granule Evaluations

The results of the flow time test meet the requirements of 10 g / sec [2]. The angle of repose test was then performed which showed that the granule of the entire formula was in accordance with the requirements of 25-45^o [10]. The flatter the angle produced, meaning the slope angle is smaller, the better the flow properties of the granule. Likewise, the results of the compressibility test for formula 1, 2, 3, 4 and 5 granules meet the requirements. Very in line with the results of the flow time test. The smaller the compressibility obtained, the better the flow characteristics.

Loss on drying test results of granules formulas 3 and 4 met the requirements of 0.4% -0.7% [11]. Formula 1, 2 and 5 do not meet the requirements. This was caused by the relative humidity of the room in the tablet manufacturing process which did not reach 25%, but only reached 40%. The greater the water content produced, it is feared that an early reaction can occur which causes the particles to become sticky with each other, thus reducing the time to dissolve the tablet.

The particle size distribution test results of formulas 2, 4 and 5 showed that many granules were distributed in sieves with number 30, while number 18 was balanced with number 60. A good graph is a normal distribution graph, which shows the results of the percentage of lagging granules in small numbers and large numbers must be balanced, while the percentage of granule distributed left in the middle number must be large. In the evaluation results of particle size distribution obtained in formulas 2, 4 and 5 in accordance with the above theory. For granule formula 1, a lot was distributed to sieve with number 20, while number 18 was balanced with number 60 and for granule formula 3, the granule was distributed to sieves with number 40, and number 18 was balanced with number 60. This occurred due to the characteristics of the granule which was not good, because if only citric acid is used in making effervescent granule tablets that are produced sticky and difficult to become granules so that when sifted will form many fines.

Table 4: The Results of Tablet Evaluations

Evaluations	F1	F2	F3	F4	F5
Color	Yellowish White	Yellowish White	Yellowish White	Yellowish White	Yellowish White
Taste	Sweet and Sour	Sweet and Sour	Sweet and Sour	Sweet and Sour	Sweet and Sour
Hardness (Kg/cm^2)	17,93	16,74	15,25	11,71	13,28
Friability (%)	0,22	0,27	0,28	0,32	0,31
Thickness (mm)	8,74	8,63	8,62	8,69	8,70
Diameter (cm)	2,55	2,54	2,54	2,54	2,54
Dissolve Time (Min)	10,50	4,52	3,32	3,13	3,42
pH	5,75	5,81	5,79	5,80	5,83

Results of Tablet Evaluations

The results of the evaluation of the weight uniformity of formula 1 to formula 5 met the testing requirements in the third edition of the Indonesian Pharmacopoeia, i.e. there were no 2 tablets having a weight deviation of 5% of the average weight and no one tablet has a weight deviation of 10% of the average weight [4].

The results of evaluation of tablet hardness obtained data exceeding the requirements of 4-8 kg/cm^2 [12]. This happens because the particle size of the granules produced is relatively small. The fine granule causes the die to clog up so that great pressure was needed to form the tablet. Great pressure causes the resulting tablet to become harder.

Furthermore, friability evaluation was carried out, from the results the friability of each formula decreases. The higher the compactness of the tablet, the smaller the friability of the tablet. For the tablet size uniformity test, it met the requirement that the tablet diameter must not be more than 3 times and not less than 1 1/3 thick of the tablet [4].

The dissolved time test was the most important parameter on effervescent tablets. This dissolved time test was observed and calculated starting when the tablet entered the effervescent medium until there was no gas bubble. In the dissolving process, effervescent tablets will produce acid and base reactions that will produce CO_2 gas. With the presence of CO_2 gas, the process of breaking the tablet will be faster and indirectly speed up the process of dissolving the tablet in water. The requirement for a good time to dissolve effervescent tablets less than 5 minutes produces a clear solution [9]. Based on the results of the study, formulas 2 to 5 met the requirements, but formula 1 did not meet the requirements. The higher the concentration of citric acid, the faster the time to dissolve the effervescent tablet, because citric acid reacts easily with sodium bicarbonate, sodium salt will form and will attract water molecules so that water will easily enter the tablet and wetted powder which will increase dissolution time, but formula 5 decreases the time dissolve. This happened because the relative humidity of the room in the process of making effervescent tablets did not reach 25%, but only reached 40% resulting in an early effervescent reaction as well as granular humidity also did not meet the requirements thus reducing the dissolution time.

The pH test was carried out to determine the degree of acidity that affects the taste of effervescent solution. From the results of the study showed the pH was at the number 5.00. This condition indicates that the tablets produced were still relatively acidic.

Analysis of data on the time to dissolve effervescent tablets. Statistical analysis uses one-way ANOVA and Tukey HSD tests. The results of normality test at dissolved time resulted in a significance value of 0.051 greater than 0.05, this indicates that the data was normally distributed. The homogeneity test results from the dissolved time data was to produce a significance value of 0.099 greater than 0.05, this indicates that the dissolved time data had the same (homogeneous) variant. The results of the analysis of variance analysis on the time to dissolve data resulted in a significance value of 0,000 smaller than 0.05, this indicates that H_o was rejected, meaning that there are significant differences from all formulas. Then the Tukey test was continued, and the results showed that there was a significant difference (<0.05) between formula 1 to formula 2, 3, 4 and 5. However there were no significant differences (> 0.05) between formula 2 and 3, formula 3 with 4 and formula 4 with 5. So, in this case formula 1 has a longer dissolving time than other formulas

CONCLUSION

It can be concluded that increasing the concentration of citric acid as a source of acid can improve the physical properties of tablets which can increase the dissolution time of tablets, while citric acid with a concentration of 35% can reduce the dissolution time of effervescent tablets dry extract of mangosteen rind (*Garcinia mangostana* L.)

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