The Effect of Variations in Citric Acid and Sodium Bicarbonate Concentrations on the Physical Properties of Effervescent Granules from a Combination of Pineapple Juice (Ananas Comosus L. (Merr)) and Dragon Fruit Juice (Hylocereus Polyrhizus)

Sukarno*, Rakhmi Hidayati, Dessy Erliani Mugita Sari, Novita Putri Wulandari

Inatitut Teknologi Kesehatan Cendekia Utama Kudus, Indonesia

*Corresponding Author: sukarnosukarno1995@gmail.com

Abstract. Pineapple and dragon fruit have good antioxidant content, so that they can be developed into more attractive preparations, one of which is effervescent granules with varying concentrations of citric acid and sodium bicarbonate. This study aims to determine the effect of variations in citric acid and sodium bicarbonate concentrations on the physical properties of effervescent granules. Method: This study is an experimental study with a combination of pineapple juice and dragon fruit juice formulated in effervescent granule preparations with varying concentrations of citric acid and sodium bicarbonate. The physical properties were then tested, including pH, water content, flow rate, angle of repose, dissolving time and Loss On Drying (LOD). Results: The results of this study showed that the three formulas met the requirements except for the examination of pH in F1 and angle of repose in F2; F3 did not meet the requirements. pH ranged from 4-7, moisture content between 0,40-1,00%, flow speed between 5,35-9,96 seconds, angle of repose 39.11-48.92, dissolving time between 3,01-1,48 seconds and Loss On Drying (LOD) ranged from 1,31- 4,4%. Conclusion: The higher concentration variation of citric acid and sodium bicarbonate affects pH, water content, flow rate, angle of repose, dissolving time and Loss On Drying (LOD).

Key words : [Pineapple and Dragon Fruit Combination, Effervescent, Citric Acid, Sodium bicarbonate]

INTRODUCTION

Indonesia is rich in biodiversity, which can be utilised for all aspects of human life, including as antioxidants. Antioxidants are compounds that can neutralise free radicals (Puspitasari et al., 2020). Antioxidants are divided into two groups based on their type: natural antioxidants, which are obtained from natural extracts, and synthetic antioxidants, which are made from chemical compounds (Rohmah et al., 2020).

The role of dragon fruit as a medicinal plant is indeed believed to be true. Its vitamin C and carbon content make dragon fruit an antioxidant (Lestari et al., 2014). The selection of red dragon fruit as an active ingredient is based on its antioxidant content, which is in line with Widianingsih's (2016) research on the antioxidant activity of methanol extract of red dragon fruit (Hylocereus polyrhizus (F.A.C Weber) Britton & Rose) resulting from maceration and concentrated by air drying, where the results obtained from the methanol extract of red dragon fruit have better antioxidant content than other types of dragon fruit. Red dragon fruit also contains many secondary metabolites such as alkaloids, tannins, flavonoids, and steroids (Jawa La et al., 2020).

Pineapple is a shrub-like fruit plant that contains vitamins A, C, calcium, dextrose, sucrose, and has a fairly high nutritional value (Egetan et al., 2016), with Vitamin C being the most dominant in pineapple. Vitamin C is useful as an antioxidant and is very good for increasing the body's immune system. Based on research conducted by Fauzana et al. (2022), pineapple has a vitamin C calibration curve with good linearity, namely a value of 144,200 mg/l.

The use of pineapple and dragon fruit needs to be made into something that is more attractive to the Indonesian people, and currently, effervescent preparations are very popular on the market. Effervescent preparations can be used as maintenance and prevention formulations (Mahdiyyah et al., 2020), one of which is available in granule form. Effervescent granules are coarse to very coarse powders containing medicinal ingredients in a mixture of acids and bases that will react when dissolved in water (Sidoretno et al., 2022). Effervescent granules have a superior appeal compared to powdered drinks in general, because they can produce carbon dioxide, providing a fresh taste like soda water and have an attractive colour, smell, and taste (Siswanto et al., 2014). Effervescent granules have two important components, namely an acid component and a base component (Rahmawati et al., 2016).

The study used citric acid as the acid component and sodium bicarbonate as the base component. According to Nursanty et al. (2022), variations in the concentration of citric acid and sodium bicarbonate affect the ideal physical properties of effervescent granules. The higher the concentration of citric acid and sodium bicarbonate given, the more significant the decrease in water content, pH, flow rate, and dissolution time. However, variations in the concentration of citric acid and sodium bicarbonate did not cause any difference in the angle of repose in the effervescent granule preparation.

METHODS

1. Making Effervescent Granules from a combination of pineapple and dragon fruit juice

   The effervescent granule preparation formula is made into 3, namely F1, F2, and F3. The difference between the 3 formulations is the variation in the concentration of citric acid and sodium bicarbonate, as shown in Table 1 below.

   Table 1. Effervescent Granule Formulation

Material	Usage indicators (%)	Amount (%)			Uses
		F1	F2	F3	
Pineapple pollen	-	20	20	20	Active substance
Dragon pollen	-	10	10	10	Active substance
Citric Acid	25-40	25	30	35	Source of acid
Sodium bicarbonate	25-50	35	30	25	Alkaline source
Aspartame	50 mg/kg	0,5	0,5	0,5	Sweetener
PVP	0,5-5	1	1	1	Fastening
Laktoce ad	-	100	100	100	Filler

   Source: Lestari et al. (2014)

   All the ingredients to be used are prepared and then weighed first according to what is needed. The dry powder of pineapple and dragon fruit that has been finely mixed with a mixture of citric acid, aspartame, some lactose and some polyvinylpyrrolidone that has been dissolved in 96% ethanol until the mass can be clenched (banana breaking). Then the mass that has been formed is sieved with a mesh sieve No. 14, then the wet granules obtained are dried in an oven at a temperature of 50°C ± 18 hours. The dried granules are again sieved with a mesh sieve No. 16 (acid component). Another container of sodium bicarbonate, some lactose and some polyvinylpyrrolidone that has been dissolved in 96% ethanol is mixed until a mass that can be clenched (banana breaking). Then the mass that has been formed is sieved with a mesh sieve No. 14, then the wet granules obtained are dried in an oven at a temperature of 50°C ± 18 hours. The dried granules are sieved again with a No. 16 mesh sieve (base component). The acid and base components are mixed and stirred until homogeneous.
2. Evaluation of Physical Properties of Granules
   a. Test pH

      A total of 5 g of effervescent granules was dissolved in 200 mL of distilled water, and then the pH was measured using a pH meter (Nursanty *et al.,* 2022).

   b. Water Content Test

      Water content testing was carried out using a moisture balance tool at a temperature of 60°C for 7 minutes (Nursanty *et al.,* 2022).

   c. Flow Rate Test

      This test was carried out by flowing 100 g of granules using a flow tester with 3 repetitions (Egetan *et al.,* 2016).

   d. Angle of Repose Test

      This test was carried out after the flow rate test by measuring the height and diameter of the pile of granules formed (Santosa *et al.,* 2017).

   e. Dissolution Time Test

      A 5 g granule of each formula was dissolved in 200 mL of distilled water. The dissolution time was measured from the time the granule was added until it was completely dissolved (Julianti *et al.,* 2022).

f. Uji Los on Drying (LOD)
Each wet granule result that will be dried and that has been dried is then weighed, and the LOD is calculated using the formula (Fatmawati *et al.,* 2020).

RESULTS AND DISCUSSION

1. Preparation of Effervescent Granules

The effervescent granule formulation of this study uses the formulation that has been done by Lestari et al. (2014) with several changes or modifications, including the use of combined active compounds from pineapple and dragon fruit and variations in the concentration of citric acid and sodium bicarbonate as acid sources and base sources. In this study, the wet granulation method was used, where the acid and base components were made separately in order to anticipate early reactions between the acid and base components. The wet granulation method is the best in making effervescent preparations because it can improve the flow properties and compressibility index (Mahdiyyah et al., 2020). The acid component consists of citric acid, aspartame, some lactose and some polyvinylpyrrolidone, which have been dissolved in 96% ethanol, while the base component consists of sodium bicarbonate, some lactose and some polyvinylpyrrolidone, which have been dissolved in 96% ethanol.

Effervescent granules are made by mixing acid and base components. They are then sieved using a 14-mesh sieve and dried in an oven at 50°C for approximately 18 hours. The 14-mesh sieve is selected to increase the number of contact points and surface area for easy drying. The dried granules are then sieved using a 16-mesh sieve. This ensures a uniform granule size.

2. Results of Evaluation of Physical Properties Test of Effervescent Granules

The purpose of testing the physical properties of the granules is to determine whether the granule preparation obtained meets the requirements and to compare which of the three variations of the formula concentration is better, so that it is hoped that it will produce a good granule preparation.

a. pH Test Results

The pH test aims to determine the acidity level of the resulting solution because it can affect the taste and freshness of the effervescent granule solution itself. If it is too acidic, it can cause and cause stomach irritation, while if it is too alkaline, it will give a bitter and less fresh taste. The pH results are said to be good if they meet the requirements with a pH value close to neutral between 5-7 (Rahmawati et al., 2016). The results of the pH test of effervescent granules of dragon fruit juice and pineapple juice can be seen in Table 2 as follows.

Table 2. Results of pH Test of Effervescent Granule Solution

Formula	First Rata – Rata	Information
F1	7,3 ± 0,69	fulfil
F2	5,6 ± 0,25	fulfil
F3	5,5 ± 0,33	fulfil

Source: Processed primary data (2024)

Description: F1: Acid: base formula (25:35)
F2: Acid: base formula (30:30)
F3: Acid: base formula (35:25)

b. Water Content Test Results

The water content test aims to determine the moisture content in effervescent granules. The amount of water in a granule can affect the stability of the granules during storage and can influence the chemical reactions of the acid and base components of the granule. A high water content can make the granules unstable, while a low water content can make the granules brittle (Anggraina, 2016). A good granule water content meets the requirements, namely less than 5% (Syaputri et al., 2023). The water content test can use two methods: the moisture balance method and the manual method by drying in an oven and then calculating it using the LOD formula. The results of the water content test of effervescent granules of pineapple juice and dragon fruit juice are seen in Tables 3 and 4 below.

Table 3. Results of the moisture balance test of effervescent granules

Formula	Water content	Information
F1	0,44± 0,036	Fulfil
F2	0,63 ± 0,041	Fulfil
F3	0,88 ± 0,115	Fulfil

Source: Processed primary data (2024)

Description: F1: Acid: base formula (25:35)
F2: Acid: base formula (30:30)
F3: Acid: base formula (35:25)

Table 4. Results of Effervescent Granule Moisture Content Test Loss on Drying (LOD) Formula

Formula	LOD	Information
F1	1,87 ± 0,50	Fulfil
F2	3,6 ± 0,2	Fulfil
F3	4,2 ± 0,15	Fulfil

Source: Processed primary data (2024)

Description: F1: Acid: base formula (25:35)
F2: Acid: base formula (30:30)
F3: Acid: base formula (35:25)

The results of water content testing using a moisture balance and LOD tool on F1-F3 showed that the stability of the substance in the granules was quite good, with LOD and moisture balance values ≤ 5%, which means the possibility of chemical and microbiological degradation reactions and product degradation is very small. Fewer acid components will have a lower water content compared to formulations that have more acid components, because citric acid is hygroscopic, so it has a greater potential to absorb water in the air, causing the humidity of the granules to increase (Forestryana et al., 2020). According to Sandrasari et al. (2016), Sodium bicarbonate can stabilise hygroscopic citric acid so that the higher the concentration of sodium bicarbonate, the lower the water content of the granules.

c. Flow Rate Test Results

The flow rate test aims to determine the flow time required for a certain number of granules to pass through a funnel or use a flow tester. A good flow rate for granules, if it meets the requirements, is one that requires no more than 10 seconds to flow 100 grams of effervescent granules (Syaputri et al., 2023). The results of the flow rate test for effervescent granules from dragon fruit and pineapple juice are shown in Table 5 as follows:

Table 5. Results of Effervescent Granule Flow Rate Test

Formula	Flow rate test	Information
F1	5,60 ± 0,22	Fulfil
F2	7,83 ± 1,15	Fulfil
F3	9,26 ±3,26	Fulfil

Source: Processed primary data (2024)

Description: F1: Acid: base formula (25:35)
F2: Acid: base formula (30:30)
F3: Acid: base formula (35:25)

d. Angle of Rpose Test Results

The angle of repose test aims to determine the good flow properties of the granules. The requirement for a good angle of repose test is ≤40° (Santosa et al., 2017). The results of the dissolution time test of effervescent granules of dragon fruit juice and pineapple juice can be seen in Table 6 as follows.

Table 6. Results of the Angle of Repose Test of Effervescent Granules

Formula	Angle of Repose	Information
F1	39,55± 0,38	Fulfil
F2	38,79± 0,48	Fulfil
F3	37,2± 0,50	Fulfil

Source: Processed primary data (2024)

Description: F1: Acid: base formula (25:35)
F2: Acid: base formula (30:30)
F3: Acid: base formula (35:25)

The results of the tests that have been carried out are in F1-F3, which obtained good results where there are results ≥ 40°, which means that they are in accordance with the applicable standards.

e. Dissolution Time Test Results

The dissolution time test is conducted to determine the time required to completely dissolve a given amount of granules, starting from the time the granules are dissolved until they are completely dissolved, indicated by the disappearance of carbon dioxide gas produced in the water. A good dissolution time for effervescent granules is less than 5 minutes (Santosa et al., 2017). The results of the dissolution time test for effervescent granules made from dragon fruit and pineapple juice are shown in Table 7 below.

Table 7. Results of Effervescent Granule Dissolution Time Test

Formula	Dissolution Time	Information
F1	3,2 ± 0,22	Fulfil
F2	2,3 ± 0,10	Fulfil
F3	1,52 ± 0,05	Fulfil

Source: Processed primary data (2024)

Description: F1: Acid: base formula (25:35)
F2: Acid: base formula (30:30)
F3: Acid: base formula (35:25)

Based on the Results of the test data obtained, it was found that all formulations met the standard requirements, namely, no more than 5 minutes. If we look at the data obtained, the concentration of acid and base does not affect the dissolution time of the effervescent granule preparation.

CONCLUSION

Based on the research conducted, it can be concluded that:

1. Variations in the concentration of citric acid and sodium bicarbonate affect the pH test, flow rate test, water content test, and dissolution time test of effervescent granules combined with pineapple juice (Ananas comosus L. (Merr)) and dragon fruit juice (Hylocereus polyrhizus).
2. A concentration ratio of 25% citric acid and 35% sodium bicarbonate has the best effect on the physical properties of effervescent granules combined with pineapple juice (Ananas comosus L. (Merr)) and dragon fruit juice (Hylocereus polyrhizus).

REFERENCES

Anggraina., & Justisia. (2016). Formulation of effervescent granule preparation of date palm (phoenix dactylifera l.) fruit extract with citric-tartaric acid as acid and effer soda as base. Jakarta.

Egetan, K. R., Paulina V.Y & Hamidah, S. S. (2016). Formulation and testing of effervescent granule preparations of pineapple (Ananas Comosus L. (Merr.)) juice. Pharmacon 5(3): 116–21.

Fauzana, A., Lesmawati., & Isna, W. (2022). Determination of vitamin C levels in fresh pineapple (Ananas Comocus L.) cultivated in Teluk Meranti sub-district, Pelalawan district using the UV-Vis spectrophotometry method. Jops (Journal of Pharmacy and Science) 5(2): 54–61.

Jawa La., Elisabeth, O., Repining, T. S., & Agustina, N. Y. (2020). Phytochemical screening and thin layer chromatography analysis of ethanol extract of red dragon fruit (Hylocereus Polyrhizus) peel. Indonesian Journal Of Pharmacy And Natural Product 3(1): 45–58.

Julianti, T., Mentari, I.A., Wikantyasning, E.R., Azzahra, S. & Hairunisa, I. (2022). Formulation and antioxidant test of effervescent granule formula of pulasan fruit peel extract (Nephelium Mutabile Blume). Jurnal Pharmascience 9(2): 287.

Lestari, P.M., Naniek, S. R., & Amalia, O. (2014). Formulation and physical evaluation of effervescent granules of dragon fruit (Hylocereus Undatus) juice. Pharmasain 2(4): 182–85.

Mahdiyyah, ., Puspitasari M. I., Putriyana, N. A., &. S yamsunarno, M. R.A. A. (2020). Formulation and evaluation of oral effervescent preparations. Pharmaceutical Magazine 5(4): 191–203.

Nursanty. R. P. (2022). The effect of variations in citric acid and sodium bicarbonate concentrations on the physical properties of effervescent granules of jambolan (Syzygium cumini L.) fruit juice. Journal of Pharmacy and Pharmacology 26(1): 38–43.

Puspitasari, A. D., Emy, S., & Ana, K. (2020). Antioxidant activity and determination of vitamin C levels of lemon (Citrus Limon (L.) Osbeck) fruit juice using the abts method. Jurnal Ilmiah Teknosains 5(2): 99– 104.

Rahmawati, I. F., Prasojo, P., & Imron, W. H. (2016). Formulation and evaluation of effervescent granules of binahong leaf extract (Anredera Cordifolia (Steen.). Pharmaciana 6(2): 139–48.

Rohmah, J., Ida, A. S., Chylen, S. R., & Devi, A. M. (2020). Antioxidant activity of ethanol, ethyl acetate, and n-hexane extracts of white turi stems (Sesbania Grandiflora (L.) Pers.) using the dpph method. Journal of Research Chemistry 5(1): 82–95.

Santosa, L., Paulina, V. Y. & Hamidah, S. S. (2017). Formulasi granul effervescent sari buah jambu mete (Annacardium Ocidental L.)." Pharmacon 6(3): 56–64.

Sidoretno, W. M., Henni, R., Indra, M., & Friskia, D. K. (2022). Formulation and evaluation of effervescent granules of a combination of dry extracts of red ginger rhizome, Javanese ginger and cinnamon. Jops (Journal Of Pharmacy And Science) 5(1): 21–35.

Syaputri, F. N. (2023). Formulation and physical characteristics test of effervescent granule preparation of ethanol extract of red betel leaf (Piper Crocatum Ruiz) as an antidiabetic. Journal of Pharmaceutical Sciences 4(1): 191–98.

Widianingsih, M. (2016). Antioxidant activity of methanol extract of red dragon fruit (Hylocereus polyrhizus (f.a.c.weber) Britton & Rose) resulting from maceration and concentrated by air drying. Jurnal Wiyata 3(2): 146–50.