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Original Paper

Smart Label with Color Indicator Made of Purple Sweet Potato (*Ipomoea Batatas* L.) on The Bottle Packaging of Pasteurized Milk

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Abstract— Smart label has made it possible to monitor and communicate information about the quality of packaged foods. Smart label is immobilized with natural dyes that are sensitive to changes in pH, namely anthocyanins. The product used in this research is pasteurized milk. The pH quality of milk will affect the storage temperature. The purpose of this study was to develop a prototype smart label of purple sweet potato's anthocyanin extract and to determine the feasibility of a smart label on the packaging as an indicator of milk freshness. Purple sweet potato extract was obtained by the maceration method using 96% ethanol and aquadest, which was acidified with acetic acid. The anthocyanin extract had a pH value of 5.60 ± 0.015 and an anthocyanin value of $70,163 \pm 0.889$ mg/100 g. The smart label shows milk freshness indicated by the changes in color. While a purple color indicates fresh milk, a faded purple color indicates that the milk is fairly fresh, and a reddish-purple smart label indicates the milk is not fresh. At room temperature storage, stale milk at 12 hours has a pH value of 5.84 ± 0.022 and a total microbe of 5.81 \log_{10} or 6.5 x 10^5 . In cold storage, stale milk on day 6 has a pH value of 5.92 ± 0.017 and a total microbe of $6.08 \log_{10}$ or 1.2 x106. The results of the feasibility of smart labels on pH stability indicate color changes in both acidic and alkaline conditions, but more stability is evident in acidic conditions.

Keywords—milk; smart label; anthocyanin extract

I. INTRODUCTION

Smart label is a label placed into a package or printed onto a packaging material to monitor product quality [1]. This smart label can provide information to consumers about product damage if it is not treated according to storage conditions. It also helps to track critical points and provide more detailed information throughout the supply chain and product distribution, helping to ensure that consumer product safety is guaranteed [26]. Product shelf life can be increased by reducing the risk of microbial contamination, biochemical and enzymatic reactions through various means such as controlling humidity and temperature, removing or reducing oxygen, adding chemical additives and preservatives, or a combination of these [2]. Packaging can extend respiration rate, delay oxidation, provide moisture, inhibit microbial growth, absorb

odors and carbon dioxide, absorb ethylene, and release aroma, for example [3]. The cellulose-acetate film can release potassium sorbate periodically, therefore serving as packaging to inhibit microbial growth. Smart labels provide information on changes in the quality of packaged materials by showing visual changes, namely the label color. Smart labels are equipped with indicators, one of which is a pH indicator.

Detection of changes in pH can use chemical dyes, but the use of chemical dyes has the possibility of a toxic effect if accidentally swallowed or in contact with the product, so it does not guarantee product safety. An alternative to overcome this problem is to use natural dyes that are sensitive to changes in pH, namely anthocyanins. Anthocyanins are red to purple colored organic compounds that can change their color in acidic and alkaline conditions. High pH anthocyanins are blue or colorless, and low pH is red [4].

Plants that have high anthocyanin content are purple sweet potatoes. Purple sweet potato's anthocyanin extract can be used to detect the freshness of milk [5].

Milk is a product that is susceptible to temperature because the milk that is stored at room temperature may decay more quickly. The acid contained in milk is mostly lactic acid. The acidity of milk is caused by various acidic compounds, such as citric acid compounds, amino acids, and carbon dioxide dissolved in milk [6]. The longer the milk is stored at room temperature, the lower the pH value will be. Fresh milk is distinguished by detecting the gas released. If milk that has decayed emits ammonia gas, it emits a gas that smells sour. Therefore, detecting stale milk can be done by measuring the increase in the acid vapor released by milk [7].

The purpose of this study was to develop a smart label prototype made of purple sweet potato anthocyanin extract. In addition, it aims to investigate the level of freshness and the feasibility of smart labels as an indicator of the level of milk freshness room temperature and cold storage.

II. MATERIALS AND METHODE

A. Materials and Equipment

Materials were pasteurized milk obtained from the dairy industry "Best Cow Farm" in Ajung district of Jember regency and purple sweet potato variety "Antin" harvested at the age of 4 months from Kaliwates district. The other materials included 96% ethanol, acetic acid, kitchen solution potassium chloride pH 1, sodium acetate solution pH 4.5, NaOH, Whatman paper no 1 paint 1001, and plastic bottles. The tools used included a hand pH meter, UV-Vis spectrophotometer, vacuum rotary evaporator, colony counter, oven, printer Canon MP 258, and ImageJ software.

B. The Preparation of Purple Sweet Potato's Anthocyanin Extract

Sweet potato anthocyanin extraction was obtained through the maceration method. Purple sweet potatoes were peeled and washed. In this study, 100 g purple sweet potato was mashed in a blender mixed with 200 ml of solvent (161 ml of 96% ethanol + 7 ml of acetic acid + 32 ml of distilled water). The purple sweet potato was macerated at room temperature for 24 hours in a closed state. The purple sweet potato extract was filtered using Whatman paper grade 41. The purple sweet potato extract was concentrated using a vacuum rotary evaporator for 20 minutes at 70°C.

C. Making Edible Film

Chitosan and CMC weighed 1.5 grams and 1 gram, respectively. Chitosan was dissolved in 100 ml of 1% acetic acid and heated using a magnetic stirrer for 30 minutes. One gram of CMC was dissolved with 100 ml of distilled water and heated using a magnetic stirrer for 30 minutes. Chitosan and CMC were mixed and heated for 15 minutes. The solution was poured into a baking dish to be compacted using an oven at 60°C for 24 hours [8].

D. Making Smart Labels

Smart labels were made of Whatman paper grade 41 immobilized with anthocyanin extract. Whatman paper grade 1 was cut in the size of 2 cm X 1 cm, soaked for 30 minutes, and dried at room temperature [5]. The layer was edible film cut with a size of 2 cm x 2 cm. The laminated paper was perforated on one side with a size of 1.5 cm x 1.5 cm. The study used laminated Whatman paper that had been immobilized with purple sweet potato's anthocyanin extract and a layer of edible film.

E. Smart Label App

Smart labels made of purple sweet potato's anthocyanin extract was applied in smart packaging to determine the level of freshness of pasteurized milk stored at room temperature (27°C \pm 3°C) and cold temperatures (5°C \pm 3°C). Pasteurized milk 100 ml/package was tested every 2 hours from the 0th hour to the 8th hour, and every 4 hours from the 8th hour to the 24th hour stored at room temperature (27°C \pm 3°C). This was performed every day for 10 days when the milk was stored at cold temperatures

 $(5^{\circ}C \pm 3^{\circ}C).$

F. The Analysis of Purple Sweet Potato

1. pH Test

Prior to the analysis, 10 ml extracts of the purple sweet potato's anthocyanin extract were taken and put into a glass beaker of 50 ml. This extract was put into the hand pH meter and observed for the pH results of the purple sweet potato's anthocyanin extract [9].

2. The Measurement of Anthocyanin Levels

Measurement of the total anthocyanin concentration of natural dyes in liquid form was carried out using the pH method differential developed by AOAC [10]. Absorbance measurement was carried out using UV-Vis spectrophotometer at wavelengths of 520 nm and 700 nm [25]. The absorbance value was calculated by the equation:

$$A = [(A520 - A700)pH 1 - (A520 - A700)pH 4.5]$$
 (1)

$$A = [(A320 - A700)pH 1 - (A320 - A700)pH 4.5]$$

$$Anthocyanin = \frac{(AXBM X FP X 1000)}{x}$$
(2)

G. The Test of Edible Film Coating

Layer tests of the edible film were conducted with a pH test. The pH test of the layer was carried out with the help of a handheld pH meter. Only 10 ml of the chitosan and CMC solution were taken and put into a glass beaker of 50 ml. It was then put into the hand pH meter and observed for the pH results [9].

H. Smart Label Characteristics

1. Color Test

The measurement was performed with the aid of image J software. The image was scanned using a printer scanner. The scan results were applied to image J and the value was determined to determine the mean of RGB [11].

2. pH Stability Test Smart label

Smart label purple sweet potato's anthocyanin extract was tested on various pH buffer solutions. Smart label at 0 hours with pH 7; 8th hour with pH 4; 16th hour with pH 1; 24 hours with pH 7 under acidic conditions and continued at 32 hours with pH 11. In alkaline conditions, smart labels demonstrate the following: pH 7 at 0 hours; pH value of 9 at 8th; pH 11 at 16th hour; pH value of 7 at 24th hour, and pH value of 4 at 32nd hour under acidic conditions.

3. Endurance Test of Smart Label Paper

The anthocyanin extract smart label paper underwent an endurance test which was carried out by soaking the smart label in distilled water for 0 hours; 12 hours; 24 hours, 36 hours, and 48 hours [24]. Drying was done using an oven for 30 minutes with a temperature of 105°C, followed by calculating weight loss.

I. Milk Test

1. pH Test

The test was measured using a hand pH meter. The pasteurized milk was only 25 ml, and it was put in the hand pH meter until the digital measurement reached the exact figure [12].

2. Total Microbial Test

The test used 25 ml milk, which was added with 225 ml of sterile 0.85% physiological saline in a 250 ml Erlenmeyer. The solution was then homogenized, resulting in a solution with a dilution of 10⁻¹. Then the test was carried out by taking 1 ml of the suspension with a 10dilution-1 with a sterile micropipette into 9 ml of sterile 0.85% physiological saline solution to obtain a dilution of 10⁻². The following phase was producing dilutions of 10^{-3} , 10^{-4} , 10^{-5} , and so on in the same way as needed. Next, 1 ml suspension from each dilution was put into a petri dish in duplicate. Afterward, 15 -20 ml sterile PCA media was added to each cup containing the suspension. For the suspension and sterile PCA media to be evenly mixed, it was important to rotate the cup forward and backward or in motion forming an 8. It was then allowed to solidify. After solidifying the dilution, it was put in an incubator at a temperature of 34°C - 36°C for 24 hours by placing the cup in an inverted position.

III. RESULTS AND DISCUSSION

- A. Development of a prototype of Smart Label Purple sweet potato's anthocyanin extract Purple Sweet Potato
- 1. pH and Anthocyanin Content

(mg/100g)

Parameter	value	SD
рН	5.60	0.015
Anthocyanin content	70 162	0.000

70.163

0.889

Table 1. Calculation of pH And Content Anthocyanin

Anthocyanin pH test demonstrates 5.60 ± 0.015 . Maceration extraction using ethanol, acetic acid, and aquadest (25:1:5) was carried out in an acidic environment, resulting in anthocyanin pH being acidic. Purple sweet potato anthocyanins are sensitive to changes in pH from acidic or alkaline conditions as indicated by changes in the color of anthocyanins.

Purple sweet potato anthocyanin content is $70,163 \pm 0.889$ mg/100 g, as demonstrated in Table 1. The type of solvent used affects the extraction yield of purple sweet potato. The anthocyanin extract is in an acidic atmosphere, while the anthocyanin pigment is in the form of flavilium cations. This will result in a greater amount of absorbance [13].

2. The pH value of the Edible Film Layer

The pH value of the layer is edible film 6.29 ± 0.015 . Film damage occurs when pH \leq 3 due to the presence of a H⁺ free which will reduce the interaction of the NH₃⁺ group with the -COO⁻ on the CMC. This interaction tends to form -COOH, which can increase its solubility with water so that the film will dissolve and become unstable. At pH>3 the film is stable, until pH 12. There is a reduction in the H⁺ group, and the bonding of the NH₃⁺ group with the -COO group⁻ tends to be stable, so that the film is not damaged at that pH [14].

B. SMART LABEL APPLICATIONS

RGB (Red, Green, and Blue) on smart labels are calculated using the aid of software imageJ.

Table 2. Values Mean RGB Smart Label

	N			
Deuteronomy	Mean of red	Mean of green	Mean of blue	Figure
1	175.652	108.303	157.454	
2	175.643	108.323	157.473	
Mean	175.648	108.313	157.464	
SD	0.080	0.014	0.013	

The value means of RGB on the smart label with the highest score is the mean of red at $175,648 \pm 0.080$, which means that red will be used in testing the intensity of color change of smart labels. The higher mean in one of the basic colors indicates that the color is the most dominantly read by the imageJ software. Every change in the average RGB shows a change in the average color intensity in the sample [15].

Smart labels are used as indicators of the freshness of milk based on changes in pH, which are indicated by changes in the color of the smart label. Pasteurized milk is packaged in plastic bottles, where smart labels are placed inside. The smart label design can be seen in Fig 1.



Fig. 1. Design of Smart label

The application of smart labels on the packaging to determine the level of freshness of pasteurized milk, which can be seen in Fig. 2.

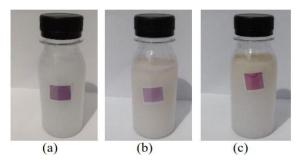


Fig. 2. Application Smart Label

Fig. 2 shows that the smart label changes color with the length of storage of milk. Figure a shows the smart label has a purple color indicating the milk is fresh. Figure b shows the smart label has a faint purple color, which indicates that the milk is fairly fresh. Figure c shows the smart label has a reddish-purple color which indicates the milk is in a stale condition or is inedible.

Changes in label color intensity occur due to changes in milk pH during the storage process either at room temperature or cold temperatures. pH range of 1-3 anthocyanins is in the form of red flavilium cations with the most stable form. When the pH rises it will cause proton loss more quickly and cause flavilium hydration [16].

- C. Freshness Level of Pasteurized Milk in Room Temperature and Cold Storage ConditionsIdentify the Headings
- 1. Pasteurized Milk Freshness at Room Temperature Storage

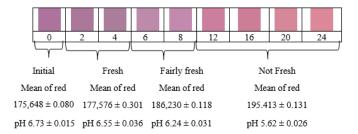


Fig. 3. Changes in color intensity of smart label corresponding to the level of freshness of milk stored at room temperature at different durations (0, 2, 4, 6, 12, 16, 20, and 24 hours)

Smart labels change color from light purple (fresh milk), faded purple (fresh milk), to reddish-purple (stale milk) at 12 hours.



Fig. 4. GrapH value of color intensity smart label at room temperature storage

The graph corresponding to the mean of the red smart label shows that the value decreases along with the length of storage of milk. The mean of the red smart label at the second hour of 176.523 ± 0.159 continues to decrease to $24\ 212,462 \pm 0.103$. These results indicate that the smart label of purple sweet potato's anthocyanin extract can be used to determine the freshness level of milk-based on visual changes in the color on the label. The lower mean of red value on the smart label indicates the lower freshness of the pasteurized milk, due to decaying milk at room temperature. The process of protein decomposition in spoiled milk causes a change in the color of

the smart label [7].

a. pH value of Milk

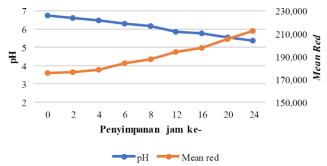


Fig. 5. The relationship between the intensity of the change in the mean of red with the pH value of milk stored at room temperature

Figure 5 shows that the pH increases during the 24-hour milk storage process at room temperature conditions. The pH value of the 0-hour milk is 6.73 ± 0.015 ; the second hour with 6.63 ± 0.035 ; 4th hour with 6.46 ± 0.037 ; 6th hour with $6.32 \pm$ 0.045; the 8th hour with 6.16 \pm 0.018; the 12th hour of 5.84 \pm 0.022; the 16th hour with 5.75 \pm 0.024; at 20 is 5.53 \pm 0.030; 24 hours with 5.35 ± 0.028 . The pH value of milk is around 6.7 [17]. If milk indicates some acidification by bacterial activity, the number will decrease significantly [18]. The factors that influence changes in the pH value of milk are sanitation, environment, disease, dilution, heating, and inaccurate measurements [19]. The acidity of milk is caused by acidic compounds. The acid contained in milk is mostly lactic acid. The acidity of milk is also caused by various acidic compounds such as citric acid compounds, amino acids, and carbon dioxide dissolved in milk [6].

b. Total Milk Microbes

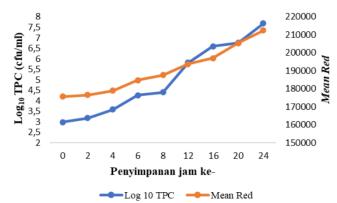


Fig. 6. The relationship between the intensity of the change in mean of red and the total microbes in milk stored at room temperature

The value of the mean of red smart label and total microbes increases with the length of milk storage. Milk kept at room temperature in the 2^{nd} hour of the smart label shows a light purple color with an average value of red 176.523 ± 0.159 and a total microbe of 2.99~log10 or $9.7~x~10^2~CFU/ml,$ which indicates the milk is fairly fresh and consumable. The 12^{th} hour shows the color of the smart label becoming reddish-purple so

that the milk can be categorized as inconsumable with a mean of red at 193.754 ± 0.129 and an increase in total microbes of $5.81 \log 10$ or 6.5×10^5 CFU/ml.

Figure 6 shows that the correlation between total microbes and changes in color intensity occurs when milk begins to rot which is characterized by an increase in the total number of microbes (TPC) and the mean of red. According to the National Standardization Agency for SNI 01-3951-1995, the maximum limit for total microbes is $3x10^4$ CFU/ml [20].

2. The Freshness of Pasteurized Milk at Cold Temperature Storage

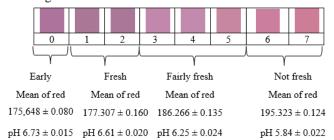


Fig. 7. Changes in the color intensity of smart labels indicating the level of milk freshness 7 days at cold temperatures (5±3°C)

The label color change categories consist of three groups, namely purple, faded purple, and reddish-purple (Fig. 7). Milk becomes inconsumable starting on day 6, which is indicated by the smart label color changing to reddish-purple.



Fig. 8. Changes in the color intensity of smart labels at cold storage

The value of the mean of the red smart label decreases along with the length of storage of milk at cold temperatures. The mean of red smart label on the first day of milk storage of 175,935 \pm 0.160 decreases to 196,660 \pm 0.141 on the 7th day. These results indicate that purple sweet potato's anthocyanin extract can indicate the freshness level of cold storage milk. The visual change in the paper color used in the smart label anthocyanin occurs due to the process of product spoilage, which is influenced by storage time and temperature [5].

a. pH value of Milk

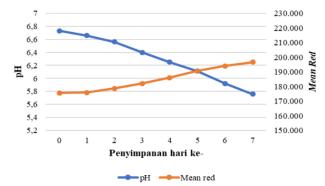


Fig. 9. The relationship between the intensity of the change in the mean of red with the pH value of cold storage milk

The pH value of milk on day 1 is 6.66 ± 0.018 and decreases until the 6th day by 6.11 ± 0.029 . The pH value indicates that the milk is still in fresh condition and still consumable. The pH value of milk on the 6th day decreases by 5.92 ± 0.017 . This pH value indicates that the milk is inconsumable.

The decrease in pH value is caused by the activity of microorganisms in milk during storage, which can cause lactose to ferment into lactic acid [18]. The smart label of extract purple sweet potato anthocyanin responds to an increase in milk pH due to the formation of volatile alkaline compounds marked by changes in the color intensity of the smart label and the mean of red is increasing.

b. Total Microbes of Milk

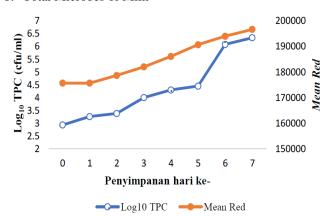


Fig. 10. The relationship between the intensity of the change in the mean of red and the total microbes of milk in cold storage

The graph indicating the mean of red on the smart label marks total microbes, which has increased with the length of storage of milk. Milk in room temperature storage on the 1st day affects the smart label to show a purple color with an average of red at 175.935 \pm 0.160 and a total microbe of 3.26 log10 or 1.8 x 10³ CFU/ml, which indicates the milk is fairly fresh and consumable. The 6th day shows the color of the smart label reddish-purple so that the milk can be categorized as rotten and inconsumable, marked with a mean of red at 196.660 \pm 0.141.

This is also indicated by an increase in total microbes of 6.08 log 10 or 1.2×10^6 .

Figure 10 shows that the correlation between total microbes and changes in color intensity occurs when the milk begins to rot, which is characterized by an increase in the total number of microbes (TPC) and the mean of red. According to the National Standardization Agency for SNI 01-3951-1995, the maximum limit for total microbes is $3x10^4$ CFU/ml [20]. Milk can be contaminated by bacteria because milk contains ingredients needed by bacteria to live such as protein, minerals, carbohydrates, fats, and vitamins. If it has been contaminated by bacteria, the state of milk can change [21].

- D. Feasibility of Smart Label of Purple sweet potato's anthocyanin extract on Packaging as an Indicator of Milk Freshness in the Field.
- pH Stability test of Smart Label of Purple sweet potato's anthocyanin extract

The pH smart label of purple sweet potato extract aims to determine the color change of smart labels in various pH conditions, both in acidic and in alkaline conditions.

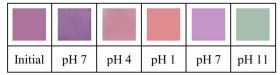


Fig. 11. Color stability of the smart label in an acidic

Figure 11 shows the difference in the intensity of the color change of the smart label of purple sweet potato's anthocyanin extract when soaked in a solution of pH 7, pH 4, pH 1, pH 7, and pH 11. Initially, the smart label of the purple sweet potato's anthocyanin extract has a light purple color. The color of the smart label soaked in pH 7 is deep bluish-purple. The color of the smart label in pH 4 at the 8th hour is light purple. The color of the smart label soaked in pH 1 at the 16th hour is reddish-purple. The smart label soaked again in pH 7 at the 24th hour has a slightly bluish-purple color, which is different from the smart label in pH 7 at the beginning. The label Smart soaked in pH 11 has a bluish color. The smart label of purple sweet potato's anthocyanin extract responds to the difference in pH solution used for soaking by showing the color change.

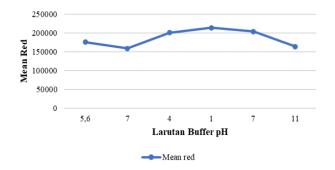


Fig. 12. The value of mean of red on the color stability of smart labels in an acid atmosphere.

The initial smart label has an average mean of red at 175.636 \pm 0.212. The value ranges across different pH values, indicative of pH 7 with 158.605 \pm 0.126, pH 4 with 200.607 \pm 0.151, pH 1 with 213.880 \pm 0.079, pH 7 with 203.748 \pm 0.167, and pH 11 with 163.530 \pm 0.245. This shows that the color of the smart label is affected by an acid solution. The lower the pH is, the higher the mean of red value becomes. The color stability of smart labels is tested under alkaline conditions.

Initial	pH 7	pH 9	pH 11	pH 7	pH 4

Fig. 13. Color stability of the smart label in an alkaline atmosphere

The initial smart label has a light purple color. The color of the smart label in pH 7 is deep bluish-purple. The color of the smart label is in pH 9. The color at the 8th hour is bluish-purple. The color of the smart label in pH 11 at the 16th hour is bluish. The smart label soaked again in pH 7 indicates a deep purple bluish color at the 24th hour. The smart label at pH 4 has a light purple color.

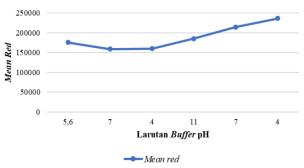


Fig. 14. The value of the mean of red on the color stability of the smart label in an alkaline atmosphere

The initial smart label has an average red value of 175.555 \pm 0.075. In pH 7 it is 158.745 \pm 0.155. In pH 9, the value is 159.895 \pm 0.125. pH 11 leads to an average of 185.112 \pm 0.102. pH 7 is 214.527 \pm 0.139, and pH 4 is 235.958 \pm 0.060. The mean of the red smart label immersed in a higher pH solution tends to decline.

The mean of the red smart label is inversely proportional to pH, meaning that the higher the pH solution used to soak the smart label, the lower the mean of red color. Smart label color changes at pH 1, pH 4, pH 7, pH 9, and pH 11, ranging from reddish-purple to bluish smart label. Smart label color change at acidic pH is more stable than at alkaline pH. The color change of anthocyanin extracts is sensitive to pH ranging from acidic pH to alkaline pH [5]. The colors of anthocyanin extracts are grouped into three pH regions, namely red, purple and blue color. Anthocyanins at low pH are in the form of red flavilium cations. When the pH is increased, it will accelerate the loss of protons to form a quinoidal base which tends to become blue or purple. In addition to increasing the pH, this causes hydration of flavilium cations to form carbinol (pseudo basic) or colorless chalcone [22].

2. Smart Label Resistance of Purple sweet potato's anthocyanin extract

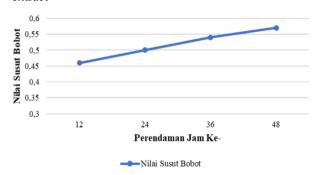


Fig. 15. Changes in the value of smart label weight loss on the length of time of immersion

The smart label weight loss value of purple sweet potato's anthocyanin extract is directly proportional to the duration of immersion, which can be seen in Figure 15. The average percentage of the weight loss of smart labels soaked for 12 hours is 0.459 ± 0.002 and further increases at 48 hours of immersion of 0.556 ± 0.001 . Weight loss is caused by water loss due to evaporation [23]. The higher the percentage of weight loss on the smart label of purple sweet potato's anthocyanin extract is caused by the loss of water on the smart label along with the length of immersion time. After soaking the smart label of anthocyanin extract for 48 hours, the smart label is still intact even though the weight loss has increased with the length of soaking time.

IV. CONCLUSION

The prototype of the smart label is developed using purple sweet potato's anthocyanin extract. The resultant label has a color that is stable in both acidic and alkaline conditions. Smart labels are made suitable for bottle packaging as an indicator of milk freshness based on pH color stability and durability of smart label paper. The freshness of milk is categorized into fresh (purple smart label color), fairly fresh (faded purple smart label color), and not fresh (reddish-purple smart label color).

NOMENCLATURE

absorbance Α A520 absorbance at a wavelength of 520 nm A700 absorbance at a wavelength of 700 nm MW molecular weight (449.2) DF dilution factor molar extinction L/cm 3 thick cuvette ι cm

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