

# USINGPOTENTIOMETRICACID- BASETITRATIONTODETERMINEP KAFROMMANGOSTEENPERICAR PSEXTRACT

*by* Nina Kadaritna

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**UTILIZANDO UMA TITULAÇÃO POTENTIOMÉTRICA DE ÁCIDO-BASE PARA DETERMINAR O pKa A PARTIR DO ESTRATO DO PERICARPO DO MANGOSTÃO**
**USING POTENTIOMETRIC ACID-BASE TITRATION TO DETERMINE PKA FROM MANGOSTEEN PERICARPS EXTRACT**

TANIA, Lisa; DIAWATI, Chansyanah; SETYARINI, Marina; KADARITNA, Nina; SAPUTRA, Andrian\*

Department of Chemical Education, Faculty of Teacher Training and Education, University of Lampung, Bandar Lampung, 35145, Indonesia

 \* Correspondence author  
 e-mail: andriansaputra@fkip.unila.ac.id

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**RESUMO**

Um dos usos dos indicadores de ácido-base é mostrar o ponto final da titulação, portanto, a determinação precisa da constante de acidez e do intervalo de pH dos indicadores precisa ser feita. Este estudo tem como objetivo determinar a constante de acidez (pKa) do extrato de pericarpo de mangostão e sua acurácia como indicador para titulação ácido-base. A determinação do pKa foi feita através do método de titulação potenciométrica simples. Os dados de titulação foram plotados em três gráficos, ou seja, pH,  $\Delta\text{pH}/\Delta V$  (o a primeira derivada) e  $\Delta^2\text{pH}/\Delta V^2$  (segunda derivada) versus volume de titulante para determinar o ponto de equivalência da titulação. comparando o volume de ácido oxálico usado para titular a solução de NaOH usando o indicador de extrato de pericarpo de mangostão e indicador de fenolftaleína. O resultado mostrou que o ponto de equivalência foi encontrado no volume de titulante de 8,6 ml e um pH medido de 9,84. o valor do extrato de mangostão foi de 7,20, e a faixa de pH foi de 6,20 a 8,20, o volume médio de ácido oxálico usado para titular 5 mL de NaOH usando fenolftaleína como indicador foi de 5,2 mL enquanto a titulação do extrato de mangostão foi 5,23 mL. A precisão do extrato de manjerição foi de 99,42%, e pode-se concluir que a titulação potenciométrica pode ser usada como uma maneira simples de determinar a constante de acidez do extrato de pericarpo de mangostão. Além disso, o extrato de pericarpo de mangostão pode ser usado como um indicador de titulação ácido-base alternativo para substituir a titulação ácido-base comum em laboratório.

**Palavras-chave:** Titulação potenciométrica ácido-base, indicador natural, constante de acidez, extrato de pericarpo do mangostão.

**ABSTRACT**

One of the uses of acid-base indicators is to show the end point of the titration, so the accurate determination of acidity constant and pH range of indicators needs to be done. This study aims to determine the acidity constant (pKa) of mangosteen pericarp extract and its accuracy as an indicator of acid-base titration. Determination of pKa was done by a simple potentiometric titration method. The titration data were plotted in three graphs, i.e., pH,  $\Delta\text{pH}/\Delta V$  (the first derivative), and  $\Delta^2\text{pH}/\Delta V^2$  (the second derivative) versus titrant volume to determine the equivalence point of the titration. The accuracy test was carried out by comparing the volume of oxalic acid used to titrate NaOH solution using the indicator of mangosteen pericarp extract and phenolphthalein indicator. The result showed that the equivalence point was found on the titrant volume of 8.6 mL and a measured pH of 9.84. so the pKa value of mangosteen pericarp extract was 7.20, and the pH range was 6.20 to 8.20. the average volume of oxalic acid used to titrate 5 mL of NaOH using phenolphthalein as the indicator was 5.2 mL while the titration used mangosteen pericarp extract was 5.23 mL. The accuracy of mangosteen pericarp extract was 99.42%. By the result, it can be concluded that potentiometric titration can be used as a simple way to determine the acidity constant of mangosteen pericarp extract. Moreover, the mangosteen pericarp extract can be used as an alternative acid-base titration indicator to substitute the common acid-base titration in the laboratory.

**Keywords:** Potentiometric acid-base titration, natural indicator, acidity constant, mangosteen pericarp extract.

## 1. INTRODUCTION

Research on acidity constant (pKa) determination from natural products has undergone rapid development. In pharmaceutical and medical research, the pKa value is important to determine the ionization of compounds in the pH atmosphere of the human (Kraft, 2003; Manchester *et al.*, 2010; Singh *et al.*, 2011). Moreover, pKa value is crucial information for drug development because lipophilicity, solubility, and permeability through membranes are very dependent on the acidity level of the drug molecules (Manallack, 2007; Manchester *et al.*, 2010; Varma *et al.*, 2012; Pahune *et al.*, 2013). In chemistry education research, researchers were trying to find and develop novel nature-based pH indicators with high accuracy compared with commercial indicators that commonly used in laboratories such as phenolphthalein, blue bromothymol, methyl orange, and others (Soltan and Sirry, 2002; Bhagat *et al.*, 2008; Shishir *et al.*, 2008; Pathade *et al.*, 2009; Patrakar *et al.*, 2010; Abugri *et al.*, 2012; Poonam *et al.*, 2012; Marulkar, 2013; Pradeep and Dave, 2013; Nikam *et al.*, 2014)

Some extracts from natural product and its corresponding pKa have been identified as indicator for acid-base titration including yellow turmeric curcumins (~8.5-10.5) (Hatcher *et al.*, 2008), bougainvillea glabra (7-10) (Gaurav *et al.*, 2010), red cabbage anthocyanin (~2.67-6.74) (Munmai & Somsook, 2011), and rose flower (2-9) (Vankar & Bajpai, 2010). One of the natural products which potentially predicted as indicators for acid-base titration is the pericarps extract of mangosteen. Mangosteen (*Garcinia mangostana*) is a fruit that thrives in Southeast Asia such as Malaysia, Thailand, Philippines, India, Sri Lanka, and Indonesia (Nazre, 2010). Extract from pericarp (peel, rind, hull or ripe) of mangosteen is known to have various medical benefits including antioxidants, antitumoral, antiallergic, anti-inflammatory, antibacterial, and antiviral activities (Pedraza Chaverri *et al.*, 2008). Components in mangosteen pericarps are xanthones, anthocyanins, proanthocyanidins, and catechins derivatives (Hiranrangsee *et al.*, 2016) with  $\alpha$ -mangosteen as the main constituent of pericarps (Won *et al.*, 2014).

Various instruments and analytical techniques have been implemented to determine pKa values including  $^{31}\text{P}$  NMR Spectroscopy (Swartz *et al.*, 2017), Capillary Zone Electrophoresis (Solow, 2006), Half Volume Method by Conventional pH Meter (Stephens &

Jonich, 1977), Isothermal Titration Microcalorimetry (Tajc *et al.*, 2004), Cyclic Voltammetry (Gooding *et al.*, 2005), the Surface-Complexation Models by Acid-Base Potentiometric Titrations (Davranche *et al.*, 2003) and etc. However, the motivation to apply simple analytical techniques with good results is needed for laboratory experiment-based chemistry learning. Then, it can be accommodated by a simple potentiometric technique.

Potentiometric titration is an easy and useful technique to determine the equivalence point on a titration thus suitable to be used for students' work in the school laboratory. Moreover, the potentiometric technique is also useful if the endpoint of the titration is difficult to be directly determined, e.g., cloudy solution or a short equivalent area. The present study focuses on the application of potentiometric acid-base titration to determine the pKa of mangosteen pericarp extract. A measured potential of solutions will be directly converted into pH according to the Nernst equation and recorded in each addition of titrant.

## 2. MATERIALS AND METHODS

### a. Visual determination of pH range

The mangosteen (*Garcinia mangostana*) pericarp used in this study was obtained from Bandar Lampung City, Indonesia. As much as 250 gram of milled pericarp was extracted using 250 mL distilled water at room temperature to obtain the extract with a concentration of 0,6 g/mL. The visual determination of pH range was done by observing the color changes when it was dropped into a solution with pH 1-14.

### b. Potentiometric titration

Potentiometric titration of mangosteen pericarp extract was carried out at 297 K by adding the standardized NaOH solution (0.204M) continuously to 50 mL of mangosteen pericarp extract until the equivalence point was reached and there was no significant increase in pH. The pH changes were recorded for each addition of 0.2 mL titrant. The pH meter used was the 911 Knick Portames pH meter No.53736/1607615/0516605 10 calibrated by NBS standard buffers (Fisher Scientific, Pittsburgh, PA), ie, pH 4.0070.01 (0.05M potassium biphthalate), pH 7.0070.01 (0.05 M potassium phosphate monobasic-sodium hydroxide), pH 10.0070.02 (0.05 M potassium carbonate - potassium hydroxide - potassium borate).

c. pKa and pH range determination

The pH data was plotted into the graph to determine the equivalence point of the titration. There were 3 graphs made, i.e., pH,  $\Delta\text{pH}/\Delta V$  (the first derivative) and  $\Delta^2\text{pH}/\Delta V^2$  (the second derivative) versus titrant volume. Determination of equivalence point was done by adding a vertical line through the three graphs. The line was started from the second derivative graph at point of  $\Delta^2\text{pH}/\Delta V^2$  was zero to the volume axis, passed the highest peak of  $\Delta\text{pH}/\Delta V$  at the first derivative graph, then passed the point where the sharp increased of pH showed. The pH and titrant volume at equivalence point was used to calculate the pKa value by the Eq. 1:

$$\text{pH} = \frac{1}{2} (\text{pKw} + \text{pKa} + \log [\text{NaA}]) \quad (\text{Eq. 1})$$

where [NaA] is equal to mole NaA/total volume. The pH range of mangosteen pericarp extract was determined by  $\text{pKa} \pm 1$ .

d. Accuracy test of the indicator

The accuracy of mangosteen pericarp extract as titration indicator was determined by using it in acid-base titration compares in the use of common indicator (phenolphthalein) as standard. The titrations were conducted by titrating NaOH solution with a standard solution of 0,1 M oxalic acid in three replication. The accuracy of mangosteen pericarp extract was determined after calculated the error percentage in using mangosteen pericarp extract by the Eq. 2.

$$\% \text{ error} = \frac{V_2 - V_1}{V_1} \times 100\%$$

Where  $V_2$  is the average volume of titrant using mangosteen pericarp extract;  $V_1$  is the average volume of titrant using phenolphthalein. The accuracy of mangosteen pericarp extract is calculated by 100%-error percentage.

### 3. RESULTS AND DISCUSSION

The experiments carried out consisted of 4 activities including: (1) pH route indicator of mangosteen peel extract visually (2) Titration of mangosteen peel extract with potentiometric titration method (3) Pricing of pKa and route of pH of mangosteen peel extract (4) Test accuracy of indicators extract of mangosteen peel.

a. Visual determination of pH range

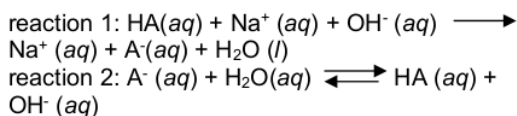
The experiment was carried out by observing the color changes of a solution in

different pH, as shown in Figure 1. At pH 1, the extract is faded pink, and the color strengthens to pH 7. Furthermore, the solution turns brown at pH 9 and gets dark brown to pH 14. The transition of color from pink to brown occurs in a pH range of 7-9 indicating pH range indicator of mangosteen extract.

The extract composed by weak acid compounds which its equilibrium is  $\text{HA} (\text{pink}) \rightleftharpoons \text{H}^+ + \text{A}^- (\text{brown})$ . According to Le Chatelier principle, the presence of HA species will be more dominant than  $\text{A}^-$  in low pH solution, and the color of the solution will be pink. Conversely, the presence of species  $\text{A}^-$  will be more dominant in a high pH solution, and the color of the solution will be brown. In the pH range 7-9, HA and  $\text{A}^-$  species are in the same amount, which gives rise to the color of a mixture of pink and brown solutions.

b. pKa and pH range determination of mangosteen pericarp extract

The initial pH of mangosteen pericarp extract was 4.34, as shown in Figure 2. It is indicated that this extract is a weak acid. At the beginning of the titration process, there is an increase in pH along with the addition of  $\text{OH}^-$  ion concentrations in solution. The reaction of the extract (HA) with NaOH solution can be written as follows:



By the addition of subsequent NaOH, there was no significant increase in pH, and this indicated the formation of a buffer solution. When the volume of NaOH added was 8.6 mL, there was a significant increase in pH. Furthermore, the next addition of NaOH, the pH value did not change significantly, and the titration was stopped at 14.2 mL NaOH addition.

Figure 2(b) and 2(c) are the first and the second derivative graph of Figure 2(a). This graph was created in order to help to determine the equivalence point of the titration. Based on the vertical line in these graphs, the equivalence point was found on the titrant volume of 8.6 mL and measured pH of 9.84. The pH at the equivalence point is more than 7 because the hydroxyl ions in the solution exceed hydrogen ions, which result from the dissociation of weak acids (HA). The NaOH volume at the equivalent point was used to determine the NaA salt

concentration, which is then used to determine the pKa value. Based on the calculation, the NaA salt concentration was 0.02994M, so the pKa value of mangosteen pericarp extract is 7.20. The pH range of an indicator is  $pK_a \pm 1$ , so the pH range of mangosteen pericarp extract was 6.20 to 8.20.

- c. Accuracy test of the indicator  
This test was aimed to calculate the

when reacted with NaOH ( $pH \pm 3$  and  $\pm 6$ ). This condition allows the use of phenolphthalein and mangosteen pericarp extract as indicators for this titration because the pH range of the two indicators includes the equivalence point of this titration. The volume of oxalic acid used in titrations was shown in Table 1.

**Table 1.** The comparison of phenolphthalein and mangosteen pericarp extract as acid-base indicators with oxalic acid used in titration

Indicators	Volume of oxalic acid 0.1 M (mL)			
	1	2	3	average
phenolphthalein extract	5.1	5.2	5.3	5.2
	5.2	5.2	5.3	5.23

Based on table 1, the average volume of oxalic acid used to titrate 5 mL of NaOH using phenolphthalein as the indicator was 5.2 mL while the titration used mangosteen pericarp extract was 5.23 mL. So the error percentage of the use of mangosteen pericarp extract as titration indicator was 0.58%. Hence, the accuracy of mangosteen pericarp extract was 99.42%. This result indicated that mangosteen pericarp extract could be used as an alternative or substitute titration indicator with high accuracy.

#### 4. CONCLUSIONS

By using a simple potentiometric acid-base titration, pKa value of mangosteen pericarp extract was 7.2 with pH range 6.2-8.2. This extract has 99.42% of accuracy as titration indicator compared to phenolphthalein. The method presented in this research is easy to be used by students in their laboratory experiment for determining pKa of other natural product. Moreover, the high accuracy of the extract indicated that this natural product could be used as an alternative for acid-base titration indicator.

accuracy of this indicator compared to indicator commonly used in the acid-base titration. The test was the titration of 0.204M NaOH solution with 0.1M oxalic acid. The indicator used in this titration is mangosteen pericarp extract and phenolphthalein with pH range 8.0 to 9.6 as standard. This process was carried out of 3 times for each indicator.

Oxalic acid has 2 equivalence points

#### 5. ACKNOWLEDGMENTS

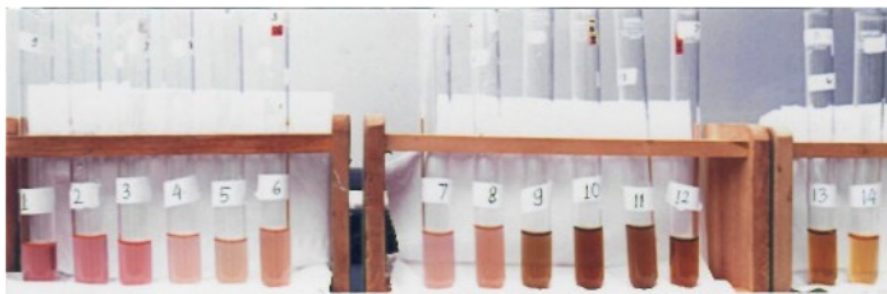
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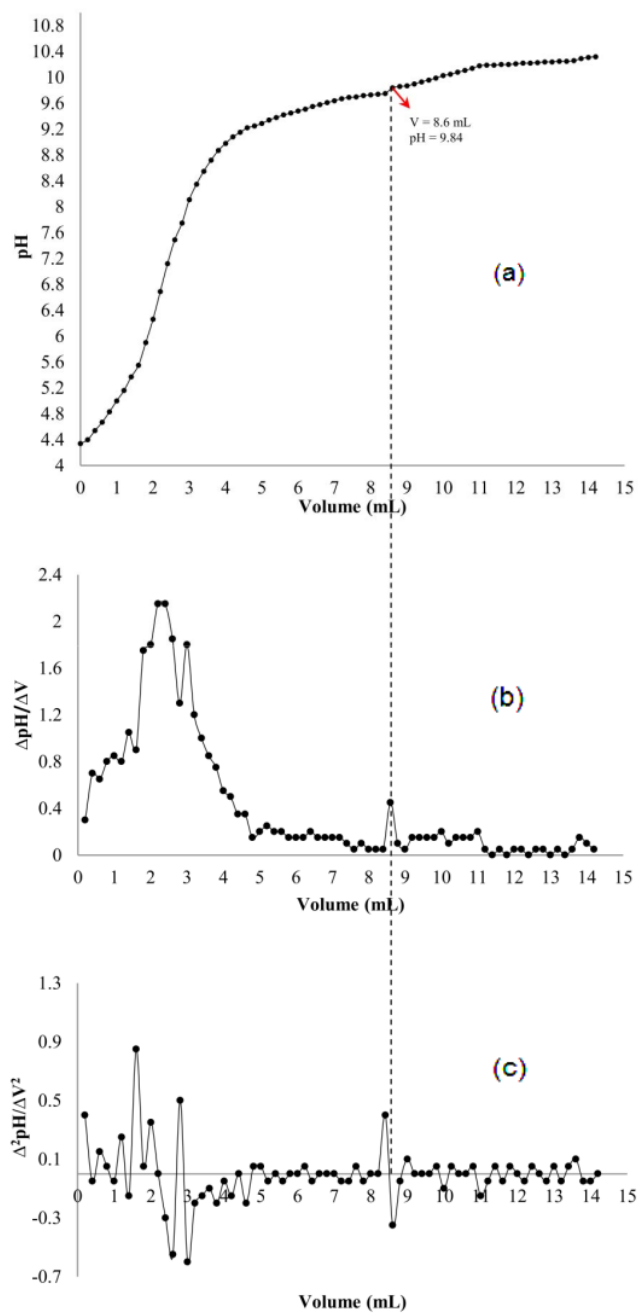
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**Figure 1.** The color of mangosteen pericarps extract in various pH solutions



**Figure 2.** (a) pH, (b)  $\Delta\text{pH}/\Delta V$  (the first derivative), and (c)  $\Delta^2\text{pH}/\Delta V^2$  (the second derivative) versus titrant volume



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