



Character Comparison Visualization of Natural Indicators in Alkalimetric Titration

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Abstract: The use of natural materials began to be developed on a large scale in chemistry. Natural substances are safe to use, easy to find, and cheaper. Biological indicators in volumetric titration are the focus of experts. This study presented a visualization of the alkalimetric titration of a weak acid with a strong base with the addition of natural indicators. *Hibiscus rosa-sinensis* L. and *Clitoria ternatea* L. flowers were used as titration indicators for diprotic acid, namely oxalic acid, with a strong base, sodium hydroxide. The phenolphthalein indicator was chosen as an indicator to compare. Maceration of flower crowns aimed to obtain biological indicator extracts. The titration was held three times for the Hibiscus flower, Clitoria flower, and phenolphthalein. The addition of flower crown extract to oxalic acid gave a pink color to the solution. There was a color change to clear when titrated. The color change indicated the endpoint of the titration. The addition of the flower crown extract indicator showed a relatively similar curve to the phenolphthalein indicator during titration. This result showed that the two flower crown extracts had the same function as phenolphthalein as an indicator for alkalimetric titration of a weak acid with a strong base.

Keywords: Alkalimetric titration, titration's indicator, *Hibiscus rosa-sinensis* L., *Clitoria ternatea* L.

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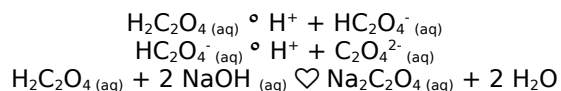
INTRODUCTION

The concentration of a chemical species can be determined by a straightforward method, namely volumetric analysis or titration. A certain amount of a solution is reacted with a standard solution of known concentration (1-3). The titration is carried out according to the solubility equilibrium (4). Titration conditions are made in such a way as to ensure that the results meet the accuracy requirements.

A virtual laboratory has been developed for titration activities. It is a very remarkable discovery. Virtual laboratories can overcome the lack of equipment when titrating in nonvirtual laboratories, improve understanding of concepts, thinking skills and motivation, and are practical

and virtually effective for online and blended learning (3-7). However, this still has the problem of what if students are faced with contextual conditions? Carrying out a titration requires good observation, patience, and vigilance. Titration activities are intended so that students are skilled in one of the laboratory activities and scientific work practices and can apply concepts or theories in chemistry practicum activities. Some of the skills expected to include measuring volume with the proper glassware, differentiating the use of glassware with different accuracy, practicing titration, processing experimental data into intelligible data, concluding experimental results, and communicating in written form.

Alkalimetric titration of a weak acid with a strong base will produce an equivalence point at $\text{pH} > 7$ due to excess hydroxide ions after the reaction.



The above reaction shows that the oxalate ion is partially ionized as a weak acid. When a solution is titrated with NaOH, a certain amount of acid is neutralized by a certain amount of base. This condition is called the equivalence point. There is an excess of hydroxide ions at equilibrium. The remaining ions cause the solution to be basic with a $\text{pH} > 7$.

Common difficulties experienced by students when doing titrations include: how to determine the right indicator for titration, how to write the following reaction equation by balancing the reaction, how to draw a titration curve, determine the type of titration, and difficulty determining the equivalence point (4,8-10). It takes the help of an indicator added in a solution that will have a striking color difference when in an acid or alkaline medium (11,12). The condition at which the indicator provides visual information is called the end point of the titration (13). The endpoint of the titration should be observed close to the equivalence point (11). Volume and pH data obtained during the titration become the basis for determining the concentration of the solution sought.

Several alkalimetric titration indicators that have been clinically tested and work over a wide range of pH can be found at chemical stores. These indicators include thymolphthalein ($\text{pH} 9.3 - 10.5$), phenolphthalein ($\text{pH} 8.3 - 10$), thymol blue ($\text{pH} 8 - 9.6$), alizarin yellow R ($\text{pH} 10.2 - 12$), cresol Red ($\text{pH} 9.3 - 10.5$). The process of synthesizing chemicals often produces chemical derivatives and other pollutants that are harmful to the environment, the cost of which is also expensive (14). Many natural materials have begun to be investigated for acid-base titration indicators by utilizing dyes found in plant parts (15). Extracts of natural compounds tend to be safe, inexpensive, and abundantly available around the environment. Natural materials that can be used as indicators include hibiscus flowers (*Hibiscus rosa-sinensis* L.), roses (*Rosa Setigera*), allamanda flowers (*Allamanda Cathartica*) (16), butterfly flowers (*Clitoria ternatea* L.) (12)(17), purple cabbage leaf (*Brassica Oleracea*) (18), batang kayu secang (*Caesalpinia Sappan* L.) (14), rosella flower (*Hibiscus sabdariffa* L.) (13)(19), red spinach (*Basella Alba*) (20).



Figure 1: *Hibiscus rosa-sinensis* L. flower.



Figure 2: *Clitoria ternatea* L. flower.

This study used hibiscus and clitoria flowers extract. Figures 1 and 2 show the flower plants. The flower crowns that are still fresh and old in color are selected for use (14). The pigments that hibiscus and clitoria flowers have are anthocyanins (12,16,17,21,22). The chemical structure of anthocyanins is shown in Figure 3. Its color range between red, violet, and blue in acidic and basic solutions. The color change is due to a change in the pH of the solution. Based on these data, hibiscus flowers and clitoria flowers can be used as indicators of alkalimetric titrations.

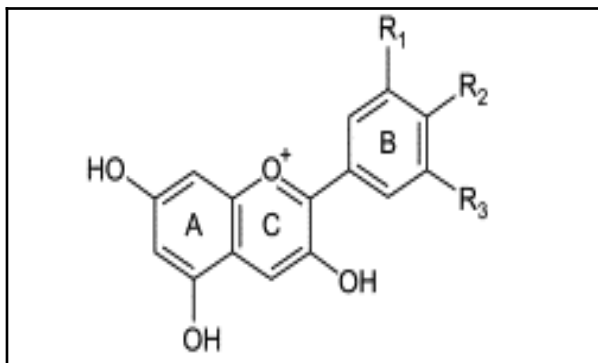


Figure 3: Anthocyanin structure.

Our study was unique because it directly compared the pH-volume curve of alkalimetric titration of a diprotic weak acid with a strong base. This study used natural indicators of *Hibiscus rosa-sinensis* L. and *Clitoria ternatea* L. flowers with phenolphthalein as a comparison indicator. Spreadsheets are used to visualize titration graphs due to their ease of use and data interpretation (23) through better graphic visualization (1,24,25). Previous research shows various flowers as alkalimetric titrations indicators by using monoprotic acid, acetic acid with a strong base, sodium hydroxide (11–13,16,17,21,22,26,27). In this study, the use of diprotic acid, oxalic acid is expected to be another alternative for selecting an acid solution for alkalimetric titration of a weak acid with a strong base.

EXPERIMENTAL SECTION

Material

0.1 M NaOH solution, 0.1 M oxalic acid solution, phenolphthalein indicator, hibiscus (*Hibiscus rosa-sinensis* L.) flower, clitoria (*Clitoria ternatea* L.) flower, ethanol 96%.

Extraction of Natural Indicators for Titration

The maceration technique carried out the extraction of anthocyanin pigments on the flower indicators of Hibiscus and Clitoria flowers (12,17,19). Hibiscus flower crowns and Clitoria flowers were chosen which have the brightest colors. Then the material was washed. Weigh 5 grams of flower crown, cut into small pieces. Put it in a dark glass bottle and soak it with 50 mL of 96% ethanol for 2 x 24 hours at room temperature in a closed state. Storage of extracts in dark bottles follows the results of previous studies (17,19).

Titration

Alkalimetric titration was performed against 10 mL of the oxalic acid solution, $H_2C_2O_4$, 0.1 M with solution NaOH 0.1 M. Three types of alkalimetric titration of a weak acid and strong were applied: 1) phenolphthalein indicator, 2) Hibiscus flower extract indicator, and 3) Clitoria flower extract indicator. The titration was held in triplicate.

Observation of the endpoint of the titration is carried out accurately. The titration produces data, namely the volume of the NaOH solution and the pH value of the neutralization reaction. We used the digital pH meter ATC 2011 to measure the pH. An excel spreadsheet describes the data and then performs a qualitative descriptive analysis.

RESULTS AND DISCUSSION

Three kinds of titrations were carried out by a strong base of NaOH against 10 mL of 0.1 M oxalic acid. Oxalic acid was used as the primary raw material because of its high purity, ease of purification, stability for a long time, strength in solution form, can be stored for a relatively long time, and has a definite relative molecular mass (28). This study chose the phenolphthalein indicator as a comparison indicator for the two natural indicators, namely the hibiscus flower and clitoria flower. Phenolphthalein was used because it has been tested as an indicator for alkalimetric titration of a weak acid with a strong base. The pink color produced by phenolphthalein indicator as a marker has ended the alkalimetric titration of a weak acid with a strong base (26,29). The addition of phenolphthalein indicator in the alkalimetric titration of oxalic acid with NaOH is shown in Figure 4.



Figure 4: The addition of phenolphthalein indicator produced a pink color at the end of the alkalimetric titration of a weak acid with a strong base.

The use of natural indicators in the identification of acids and bases has begun to be developed because they are easy to obtain, safe, and have cheaper production costs, and the results are no different when compared to the use of artificial indicators (29–31). The titration in this study used flower extract indicators that are easily found in the environment, namely Hibiscus flowers and Clitoria flowers. Extracts of both flower crowns could produce color changes when interacting with acids or bases. They are shown in Figure 5. A good indicator is an indicator that has a pH change trajectory around the equivalence point or at the

equivalence point and can show clear and sharp color changes (11,26).

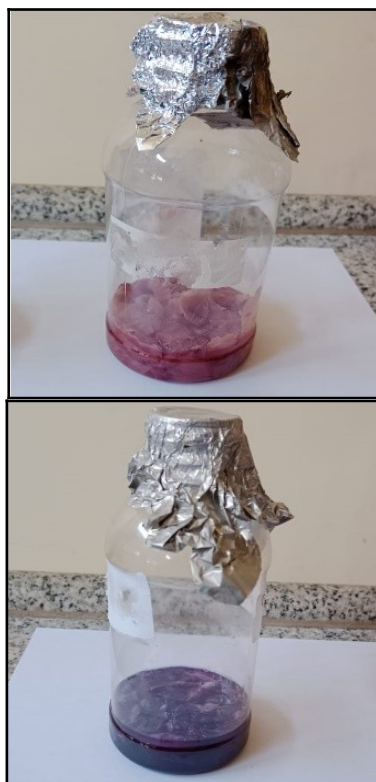


Figure 5: The result of maceration of hibiscus (top) and clitoria (bottom) flower crowns.

Hibiscus and butterfly flower crowns contain anthocyanin pigments. Both hibiscus and clitoria flowers indicator gave a pink color when interacting with weak acids. The color slowly faded after titration with a strong base. Figure 6 shows the results of the titration of oxalic acid with NaOH using the hibiscus indicator and clitoria flowers.

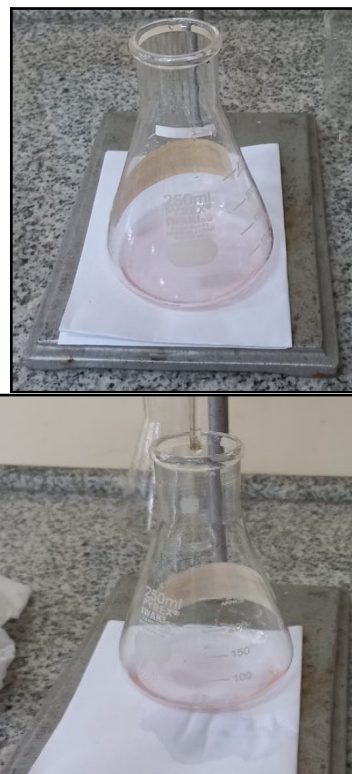


Figure 6: Changes in the color of the solution after titration due to the addition of hibiscus (top) and clitoria (bottom) flowers indicators.

This study compared the visualization of alkalimetric titration results using natural indicators of flower crown extract against Phenolphthalein as an indicator. Titration used a weak diprotic acid in which there are two values of K_a . Oxalic acid used in this titration undergoes double ionization in the solution, resulting in the values of $K_{a1} = 5.6 \times 10^{-2}$ and $K_{a2} = 5.4 \times 10^{-4}$. These two values of K_a explain that oxalic acid has the ability to produce two hydrogen ions per molecule. While the strong base NaOH is only able to produce one hydroxide ion per molecule. Thus, when NaOH was titrated with oxalic acid, two hydrogen ions were equivalent to 1 mole of hydroxide ions. Or in other words, two hydrogen ions from oxalic acid were exactly neutralized by one hydroxide ion from NaOH.

The results of the study obtained that the alkalimetric titration data of a strong base NaOH against a weak diprotic acid, oxalic acid using indicators namely phenolphthalein (P), hibiscus flower (H), and clitoria flower (C), are presented in Table 1.

Table 1: Titration pH and NaOH Volume.

Vol NaOH (mL)	pH (T1-P) 1	pH (T2-P) 2	pH (T3-P) 3	pH (T2-H) 5	pH (T3-H) 6	pH (T1-C) 7	pH (T2-C) 8	pH (T3-C) 9
0	1.72	1.72	1.71	1.69	1.7	1.57	1.57	1.6
2	1.78	1.77	1.78	1.9	1.9	1.69	1.67	1.74
4	1.95	1.88	1.85	2.03	2.01	1.7	1.86	1.86
6	2.11	2.08	2.07	2.07	2.09	1.79	1.9	1.99
8	2.48	2.57	2.42	2.16	2.47	1.9	2.05	2.09
10	3.05	3.19	3.12	2.67	2.83	2.02	2.47	2.39
12	3.46	3.74	3.64	3.12	3.12	2.47	2.89	2.83
14	3.87	4.05	3.96	3.47	3.52	2.91	3.23	3.29
16	4.5	4.9	5	3.8	3.79	3.21	3.51	3.52
18	9.77	9.77	10.13	4.13	4.17	3.52	3.77	3.79
20	10.5	10.12	10.87	4.87	4.86	3.77	4.17	4.17
22	10.81	10.61	10.94	9.88	9.89	5.2	5.22	5.22
24	10.88	10.66	10.96	10.12	10.12	9.72	9.72	9.71
26	10.98	10.75	10.96	10.3	10.3	10.25	10.26	10.12
27	11	10.96	11	10.42	10.4	10.4	10.26	10.28

Visualization of the alkalimetric titration curve for a weak acid and a strong base was performed using an excel spreadsheet, presented in Figure 7. The graph of the alkalimetric titration results through a spreadsheet application provided convenience in

data interpretation. The interaction of a volume of acid with a base produced plots of pH values. The graph was identical to the graph of an alkalimetric titration of a weak acid with a strong base, which is in the form of an S.

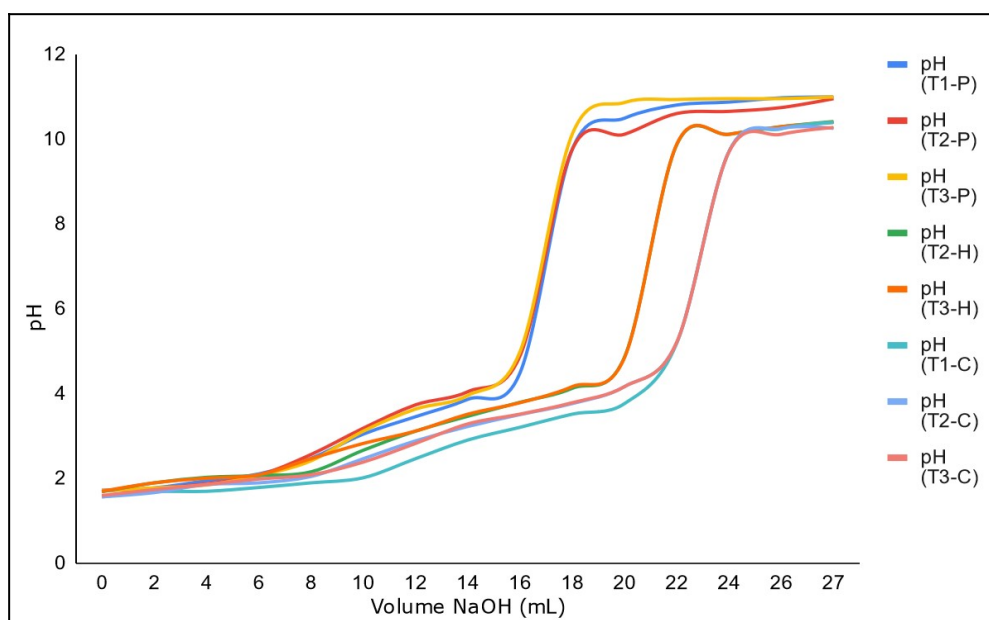


Figure 7: Alkalimetric titration (T) with phenolphthalein (P) indicator, hibiscus (H) flower and clitoria (C) flower.

Data were obtained from first to ninth titrations. Data for alkalimetric titrations with phenolphthalein indicator were displayed in data first to third.

Alkalimetric titrations with the hibiscus flower indicator were established in the fourth, fifth, and sixth data. The fourth data revealed an erroneous

result, not so for the fifth and sixth data. The fifth and sixth data indicated relatively the same results. The clitoria flower indicator's titration was shown in the seventh, eighth, and ninth data.

The fourth data had the most different values from the fifth and sixth data. It could be caused by human error that occurred during the titration. When carrying out titration, some things that must consider include preparing the equipment correctly. Glassware must be completely clean of impurities. Pay attention to the burette used for the titration. Check the readiness of the burette. Replace if the burette is cracked, broken, or leaks at the faucet or valve. Next is to prepare the materials used, namely titrant and indicator. Use standard solutions and be free from other impurities. Prepare yourself before doing the

titration. Use personal protective equipment. Confidently perform the titration, not in rush, but thorough. Pay attention to the addition of the titrant volume and pH value when you carry out the titration.

Oxalic acid reacted with the addition of NaOH shows a graph trend that increases from pH 2 to pH 4. Table 2 presents graphs, line equations, and coefficients of determination for the entire titration using phenolphthalein, hibiscus, and clitoria flowers as indicators. A strong coefficient of determination shows that the change in pH value is correct due to adding of a certain amount of NaOH. It indicates that the titration has been carried out correctly. There is no significant difference between the three gradients of the line equation.

Table 2: Graph of Oxalic Acid with NaOH Reaction at pH 2 to pH 4.

Figure	Indicator
	<p>phenolphthalein $y = 0.2639x + 0.4426$ $R^2 = 0.9881$</p>
	<p>hibiscus $y = 0.1915x + 0.8311$ $R^2 = 0.9871$</p>

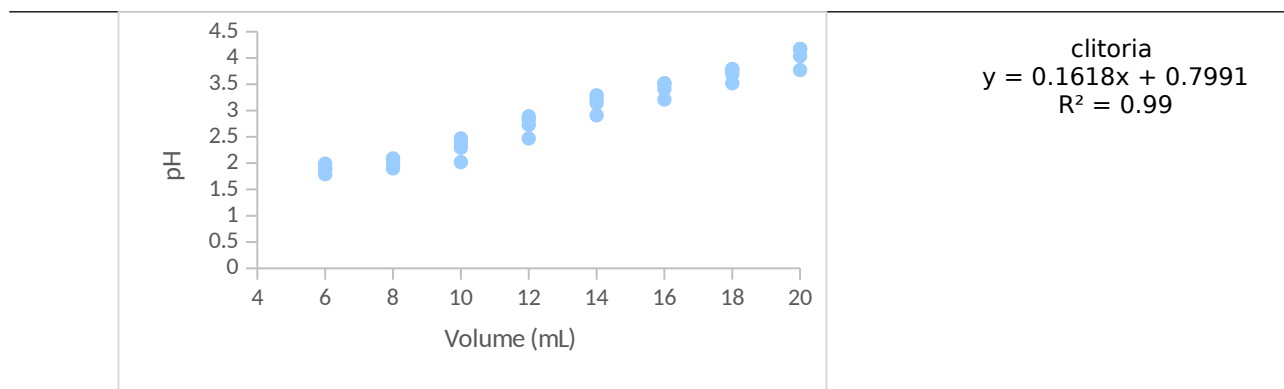


Table 2 provides information about alkalimetric titration using phenolphthalein indicator. The addition of NaOH volume from 16 mL to 18 mL showed a significant increase in the pH value, namely pH 4.5 - 5 to pH 9.77 - 10.13. In the alkalimetric titration using a hibiscus indicator, there was a significant change in the pH value from pH 4.86 to pH 9.89 for the addition of NaOH volume from 20 mL to 22 mL. As for the titration with the clitoria flower indicator, the change in pH 5.22 to pH 9.72 was indicated by the addition of NaOH volume from 22 mL to 24 mL. Such conditions implied that the titration was approaching the equivalence point. The pH value rose faster at first, but the increase became slower until it approached the equivalence point.

The equivalence point of the titration indicated that a certain amount of acid had been neutralized by the addition of a certain amount of base. Visualization of the pH equivalence point of the titration of a weak acid with a strong base was not correct in 7. The effect of the hydroxide ion possessed by the strong base used NaOH gives a pH value of > 7. There were still residual hydroxide ions when equilibrium occurred. The pH value increased more rapidly at the equivalence point. The three types of titrations produced almost the same equivalence point pH values, namely in the pH range of 9.72 - 9.89. This event follows the fact that the equivalence point of the alkalimetric titration of a weak acid with a strong base is at pH 8 - 10 (11,21).

In the pH range of 4.13 - 5.22, the color of the solution began to change in the three titrations. The pink color disappeared and then reappeared. And in the pH range of 9.71 - 10.13, the color of the solution no longer changed. Titration with phenolphthalein, the color of the solution changed from clear to pink. Meanwhile, in the titration with the indicator, both the Hibiscus flower and the Clitoria flower changed color from pink to clear. This indicated that the endpoint of the titration had occurred. It was advisable to stop the titration immediately.

In general, the increase in the value of pH 9 to pH 10 occurred very slowly, even though repeated

amounts of NaOH were added. This slow increase could be explained because the solution formed a buffer system. Buffer solutions are produced by adding a small amount of a strong base to a weak acid. The buffer solution will maintain its pH value to a certain extent (2,11,15). At pH 10.12, the solution was even more difficult to raise the pH value. In this condition, there was an excess of hydroxide ions after equilibrium, so the pH of the solution became very basic.

CONCLUSION

The extraction of the hibiscus flower and clitoria flower could be obtained by curing the flower crown for 48 hours using ethanol. The extract obtained could be used as a titration indicator because it gave a clear and sharp color change. The pink color appeared when the flower crown extract was added to the oxalic acid solution. The endpoint of the titration was indicated when the pink color had changed to clear. The pH trajectory produced when the hibiscus flower and clitoria flower indicators work had similarities with the addition of phenolphthalein which had been tested as an indicator for alkalimetric titration of a weak acid with a strong base.

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