



Analytical Potentials of Dye Extracts from *Urena Lobata* (Mgbo) Flowers

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Abstract. Indicators used in titrations show well-marked changes of color in certain intervals of pH. Most of these indicators are organic dyes and are of synthetic origins. The present study was designed to evaluate the extracts of a flower from a plant that is easily available. The analytical potentials of ethanol, cold water, and hot water extracts of dyes from the flowers of a common plant, *Urena lobata* were evaluated. 3 drops of the dye extract were dropped in 10ml of 0.1M H₂SO₄. This was titrated against 0.1M NaOH until the colour changed from light pink to light orange at equivalent point. The titration was repeated using standard indicators such as phenolphthalein, methyl orange, bromothymol blue and methyl red for weak acid strong base, strong acid strong base, strong acid weak base and weak acid weak of equimolar concentrations. The same acid and base of same strength were also assessed using potentiometric titration. These results indicated that flower extracts of this plant can be used as acid-base indicator in all types of titration. The pH of these indicators was also determined. The rationale behind using these natural indicators in preference to synthetic indicators is its easy availability, inertness, ease of preparation and cost effectiveness.

Key Words: *Urena lobata*, acid base titration, potentiometric titration, pH, natural indicator.

INTRODUCTION

Commercial indicators are expensive and some of them have toxic effects on users and can also cause environmental pollution (Pathade, 2009). For these reasons there has been an increasing interest in searching for alternative sources of indicators from natural origins. These alternatives would be cheaper, more available, simple to extract, less toxic to users and environmentally friendly.

Volumetric analysis is one of the key quantitative techniques used to analytically determine both inorganic and organic acid interaction with strong or weak acids and bases in raw materials, intermediates and finished products for quality assurance purposes. This is accomplished via the use of appropriate weak organic dyes or acids pH indicators. Most pH indicators are either weak organic acids or basic dyes which accept or donate electrons. The change in color at a marginal range is attributed to their acidic or basic properties. Some inorganic compounds such as potassium bromate and iodates are also used in analytical chemistry (Housecroft and Sharpe, 2008)

Although there are automated titration apparatus that determine the equivalent points between reacting species, indicators are still needed for teaching and research laboratories for simple titrations (Nwosu, 2004). Natural indicators have been extracted from Hibiscus (red species), Bougainvillea and rose flowers (Nwosu, 2004).

Several authors have reported the effectiveness of natural indicators in acid-base titrations e.g. *Nerium odorum*, *Thespesia populnea* extracts used as indicators (Patil, 2009); *Morus alba linn.* fruit extracts indicator (Pathade, 2009) and *Ixora coccinea*, *Datura stramonium*, Sun flower (*Helianthus annus*), pride of Barbados (*Caesalpinia pulcherrima*) and rail creeper (*Ipomoea palmate*) flower petal extracts (Nwosu, 2004). The natural indicator sources investigated in these papers have been extracted and prepared using ethanol, hot water, or cold water. Eze and Okereafor, 2002 also investigated the industrial and analytical

potentials of dye extracts from the fruit of *Telfrair occidentalis* Eze and Okerefor, 2002 also investigated the industrial and analytical potentials of dye extracts from the fruit of *Telfrair occidentalis*. On a previous study carried out by Azundo 2006 (Unpublished data), dyes were extracted from the guinea corn leaves and used for titration. However, the data was not consistent and little is known about whether the dyes could react well with all types of reacting species (strong bases versus strong acids and or weak acids versus strong base or the vice versa or weak acids versus weak bases). Also no evidence is available with regards to the pH ranges of the indicator, its optimum function and its possibilities of replacing some expensive commercial indicators. Unlike some commercial indicators that are known to have detrimental effects, we anticipate that indicators from natural sources could reduce both environmental pollution and the toxic effect on users. This will also encourage the cultivation of the crop in large scale for multipurpose uses.

A pH indicator is a halochromic chemical compound that is added in small amounts to a solution so that the pH (acidity or alkalinity) of the solution can be determined easily. . An acid- base indicator is usually a weak organic acid denoted as HIn that has a different colour than its conjugate base (In^-) (Silberberg, 2006). Hence a pH indicator is a chemical detector for hydronium ions (H_3O^+) (or Hydrogen ions (H^+) in the Arrhenius model). Normally, the indicator causes the color of the solution to change depending on the pH. An indicator changes colour over a range of pH (Ikoku *et al*, 1984)In this study we observed the reaction of flower extract in different pH conditions and compared natural indicator to commercial indicators with measurement of pH.

MATERIALS AND METHODS

The chemicals and materials used for the study include; ethanol, cold water, hot water, and reagents including sodium hydroxide (NaOH), Sulphuric acid (H_2SO_4), Oxalic acid (COOH)₂2H, Sodium hydroxide (NaOH), Amonium hydroxide (NH_4OH)whih were of analytical grade and were used without further purification,

methyl red, phenolphthalein, methyl orange, Bromothymol blue, flower extracts of *Urena lobata*, and 6 filter papers. The following apparatus such as; pH meter, volumetric flasks (100 ml, 500 ml and 1000 ml), conical flask with volume size of 50 ml, burette of 50 ml, and graduated measuring cylinders of volume size 10, 50, 100 and 500 ml were used to carry out the experiment. The analytical grade reagents were made available by the Department of Pure and Industrial Chemistry, Abia State University, Uturu, Nigeria.

Sample Collection and Preparation of Indicator Solution

Urena lobata flowers were collected from plants growing wild in bushes in Okigwe, Imo State, Nigeria. The flowers were collected and were kept at room temperature. They were dried to minimize oxidative loss before pounding into fine powder with mortar and pestle. The resulting powders were sieved. The natural indicator extract was prepared by weighing approximately 2 g of a powdered sample flowers into 50ml beaker and 20.0 ml of ethanol, hot water, and cold water was added. The mixture was vortexed for 5 minutes at ambient temperature (25°C) and then filtered using Whatman No. 1 filter paper into a new culture test tube of (20 × 250 mm), capped with a Teflon cap and store for use on the same day.

The flowers were collected and were kept at room temperature. They were dried to minimize oxidative loss before pounding into fine powder with mortar and pestle. The resulting powder was extracted with ethanol, hot water and cold water. Finally the extract was filtered and used as indicator.

The experimental work was carried out by using the same set of glass wares for all type of titrations. As the same aliquots were used for both titrations i.e. titration by using standard indicators and flower extract, the reagent were not calibrated. The equimolar titrations were performed using 10 ml of titrant with three drops of indicator. All the parameters for experiment are given in **Table 1.1**. A set of three experiments was carried out and mean and standard deviation were calculated from results.

Experimental Procedures

Experiment with Natural Indicators

Approximately 10.0 ml of 0.1 M H_2SO_4 or 0.1 M $(\text{COOH})_2\text{H}_2\text{O}$ was titrated with 0.1 M NaOH using the dyes, extracted from the Urena lobata in the order of strong acid versus strong base and weak acid versus strong base respectively, and then 10.0 ml of 0.1 M H_2SO_4 or 0.1 M $(\text{COOH})_2\text{H}_2\text{O}$ was also titrated against the weak base 0.1 M NH_4OH in the order of (H_2SO_4 v/s NH_4OH , $(\text{COOH})_2\text{H}_2\text{O}$ v/s NH_4OH). Three drops of the extracted indicator were added to each volume of acid used for the titration. The experiment was conducted in triplicate as indicated in **Table1.1**. The acid-base titration was carried out at room temperature.

Experiment with standard Indicators

For comparison, the procedure used for the commercial indicators (standard indicators) was the same as described above for the natural indicator. The experiment was conducted in triplicate and the results were analyzed with simple Microsoft excel 2010 and SPSS statistical software. The statistics generated were used to discuss the results.

Potentiometric titration with natural indicators

The pH meter was calibrated to 4.0, approximately 10.0 ml of 0.1 M H_2SO_4 or 0.1 M $(\text{COOH})_2\text{H}_2\text{O}$ was titrated with 0.1 M NaOH using the natural indicator, extracted from the Urena Lobata in the order of strong acid versus strong base and weak acid versus strong base respectively, as the pH of the acid was recorded before titrating. The base (NaOH) was being added in 1ml each as the pH reading is being recorded, at a point there will be a sudden change in pH and color change. The titration continues with 1ml each, until there is little or no change in pH. And then 10.0 ml of 0.1 M H_2SO_4 or 0.1 M $(\text{COOH})_2\text{H}_2\text{O}$ was also titrated against the weak base 0.1 M NH_4OH in the order of (H_2SO_4 v/s NH_4OH , $(\text{COOH})_2\text{H}_2\text{O}$ v/s NH_4OH), as the pH of the acid was recorded before titrating. The base (NH_4OH) was being added in 1ml each as the pH reading is being recorded, at a point there will be a sudden change in pH and color change. The titration continues with 1ml each, until

there is little or no change in pH. Three drops of the extracted indicator were added to each volume of acid used for the titration. The result of this experiment is shown in chapter four of this work.

Potentiometric titration with standard Indicators

For comparison, the procedure used for the commercial indicators (standard indicators) was the same as described above for the natural indicators. The results were analyzed with simple Microsoft excel 2010 and SPSS statistical software. The statistics generated were used to discuss the results.

RESULT AND DISSCUSION

pH Range of Indicators: The results of the titre values obtained using the natural indicator (Urena Lobata extracts) and the standard indicators are presented in **Table 1.1**

Table 1.1. Mean volume (in ml) of base used to reach the equivalent point of acid-base titrations.

Reaction Type	H ₂ SO ₄ /NaOH	(COOH) ₂ H ₂ O /NaOH	H ₂ SO ₄ / NH ₄ OH	(COOH) ₂ H ₂ O / NH ₄ OH
Indicator Type	Vol(ml)	Vol(ml)	Vol(ml)	Vol(ml)
Ethanol Extract	22.6 ± 0.17	20.7 ± 0.17	4.2 ± 0.2	1.40 ± 0.30
Hot water extract	23.3 ± 0.12	20.5 ± 0.17	3.6 ± 0.1	3.2 ± 0.1
Cold water extract	23.3 ± 0.08	21.3 ± 0.14	4.2 ± 0.17	3.5 ± 0.24
Bromothymol blue	23.2 ± 0.20	20.6 ± 0.20	3.7 ± 0.20	3.4 ± 0.25
Methyl Red	22.7 ± 0.11	21.2 ± 0.16	3.8 ± 0.4	3.2 ± 0.20
Methyl Orange	21.2 ± 0.16	19.9 ± 0.1	3.8 ± 0.1	3.3 ± 0.1
Phenolphthalein	23.1 ± 0.1	23.3 ± 0.2	4.0 ± 0.1	3.7 ± 0.14

Concentration of H₂SO₄ = 0.1 M, NaOH = 0.1 M, (COOH)₂H₂O = 0.1 M, and NH₄OH = 0.1 M.

For the titration of strong acid strong base ($\text{H}_2\text{SO}_4/\text{NaOH}$), It can be seen that the extracts in ethanol, hot and cold water gave an average end point of 23.07cm^3 . However, The end point of the ethanol extract 22.6 ± 0.17 was closest to that of methyl red (22.7ml) and so can effectively replace it in acid base titrimetry for strong acid/strong base. There was also no significant difference between the cold and hot water extracts and their end points matches that of bromthymol blue 23.2 ± 0.20 and phenolphthalein (23.10 ± 0.1). So the cold or hot water extracts of *Urena lobata* can effectively replace the commercial synthetic organic indicators bromothymol blue and phenolphthalein in acid base titrations using strong acid strong base.

For weak acid strong base ($(\text{COOH})_2 \cdot 2\text{H}_2\text{O}/\text{NaOH}$) the average end points for the three extracts in ethanol. Cold and hot water is 20.83ml with the ethanol extract end point (20.2 ± 0.17 being closest to that of bromothymol blue (20.6 ± 0.20)). For strong acid/weak base ($\text{H}_2\text{SO}_4/\text{NH}_4\text{OH}$) and weak acid/ weak base, the end points were significantly very smaller 3.6 ± 0.1 - 4.2 ± 0.2 and 1.40 ± 0.30 - 3.7 ± 0.14 respectively than those of the strong acid/ strong base and weak acid/ strong base 21.2 ± 0.016 - 23.3 ± 0.12 and 19.9 ± 0.1 - 23.3 ± 0.2 respectively for titration using the dye extracts as indicators as well as the commercial indicators. The ethanol and cold water extracts can effectively replace phenolphthalein for weak acid /weak base analysis while the hot water dye extract can replace methyl red as both gave the same end point 3.2ml for weak acid weak base.

Colour changes of the Indicators Used in The Titration

The indicator colour changes of the dye extracts as well as the commercial indicators is shown in table 2

Table 1.2. Physical and chemical parameters of the indicators used for titration

Acid	Base	Indicator color change and (pH range) (Extract)			Indicator color change and (pH range) (Standard)			
		Ethanol extract	Hot H ₂ O extract	Cold H ₂ O extract	Methyl orange	Phenolphthalein	Methyl red	Bromothymol blue
H ₂ SO ₄	NaOH	Light pink to light lemon green (0.74 – 9.09)	Light pink to light lemon green (0.80- 10.92)	Light pink to light lemon green (0.79- 5.15)	Reddish to yellow (0.26 – 6.12)	Colorless to pink (0.19 – 11.82)	Light pink to orange (0.11 – 10.60)	Yellow to blue (0.14 – 11.72)
(COOH) ₂ H ₂ O	NaOH	Light pink to light lemon green (0.97 – 12.49)	Light pink to light lemon green (0.23 - 11.5)	Light pink to light lemon green (0.19 – 11.05)	Reddish to yellow (0.26 – 12.07)	Colorless to pink (0.23 – 12.31)	Light pink to orange (0.21 – 11.50)	Yellow to blue (0.19 – 12.31)
(COOH) ₂ H ₂ O	NH ₄ OH	Light pink to light lemon green (0.19 – 8.89)	Light pink to light lemon green (1.03 – 10.04)	Light pink to light lemon green (0.89 – 8.60)	Reddish to yellow (0.99 – 4.71)	Colorless to pink (1.08 – 9.62)	Light pink to orange (0.00 – 8.11)	Yellow to blue (0.86 – 7.68)
H ₂ SO ₄	NH ₄ OH	Light pink to light lemon green (0.86 – 8.68)	Light pink to light lemon green (0.17 – 7.96)	Light pink to light lemon green (0.21 – 6.42)	Reddish to orange (0.25 – 6.54)	Colorless to pink (0.41 – 6.05)	Light pink to orange (0.51 – 7.56)	Yellow to blue (0.47 – 8.26)

It was observed that the natural indicator (*Urena Lobata* extracts) when added to the acid produced a light pink color as. The pH ranges of the indicators is also shown in table 3. A very useful information from the pH measurements is that the ethanol extract of *Urena lobata* has the some ph range with phenolphthalein (12.84-13.28). It was observed that the pH range of the acid was affected by the indicator added because of the nature of the indicators. Methyl red has the pH range of 1.14 – 2.77 due to its acidic nature it reduces the pH of the acids most even to 0.00 like in Oxalic vs NH_4OH . The possible factors that might have contributed to the pattern of the pH variation as well as titre value could be temperature, ionic strength, colloidal particles and organic solvents (Skoog, 1994).

Table 1.3. The pH range of both the natural and synthetic indicators.

Indicator type	pH range	Color change
Ethanol extract of <i>Urena Lobata</i>	12.84 – 13.28	Light pink to light lemon green
Hot water extract of <i>Urena Lobata</i>	11.05 – 13.28	Light pink to light lemon green
Cold water extract of <i>Urena Lobata</i>	11.05 – 13.28	Light pink to light lemon green
Phenolphthalein	12.84 – 13.28	Colorless to pink
Methyl Orange	1.14	Reddish to yellow
Methyl red	1.14 – 2.77	Light pink to orange
Bromothymol Blue	6.87 – 13.21	Yellow to blue

Equivalent Points: The average titre values obtained for the extract was comparable to methyl orange, phenolphthalein methyl red and bromothymol blue indicator used as indicated in **Table 1**. It was interesting to observe that for the weak acid versus strong base the titer values of cold water extract of *Urena Lobata* was similar to that of methyl orange while all other extract are similar with phenolphthalein, methyl red and bromothymol blue. A similar trend was found in strong acid versus weak base but phenolphthalein is similar to that of methyl orange and cold water extract of *Urena Lobata*. For weak acid versus weak base, the methyl orange were quite different to the natural indicator extract and synthetic indicator, as presented in **Table 1**.

Conclusion

The analytical potentials of the ethanol, cold and hot water extracts was successfully examined, The analysis show that the plant extracts can effectively replace the commercial indicators for different types of acid-base titrimetry and so will be very useful considering safety and cost.

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