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Acid-Base Indicator Properties of Dye from Local Plant: The Rosella Calyces (*Hibiscus Sabdariffa*)

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Abstract

The dye from the Calyces *Hibiscus Sabdariffa* (Zobo) was extracted and used as acid-base indicator for the standardization of acid and determination of the molar masses of some selected acids. The Calyces *Hibiscus sabdariffa* was peeled, washed and heated in an oven at 60°C. It was ground into powder and soaked in hot and cold ethanol for the extraction. Part of the extract was filtered and concentrated by subjecting the extract to slow heating which yielded 1.5 kg of the Hibiscus sabdariffa indicator. Hot and cold extracts of the *Hibiscus sabdariffa* were used for the standardization of acid with 0.03 M concentration of the bases. On the preliminary test carried out on basic medium, the calyces *Hibiscus sabdariffa* indicator turned red in acidic condition and green in basic medium, both hot and cold indicators yielded sharp and intense colours at the end points during titration processes. However, there were no colour changes for weak acid versus weak bases ($H_2C_2O_4$/Na_2CO_3 and $H_2C_2O_4$/$B_4Na_2O_7$). The mean volumes of the acids used were determined and used for the determination of the molar concentration and mass concentration of all the acids used in the research work.

Keywords: Indicator • Acids • Bases • *Hibiscus sabdariffa* • Rosella Calyces • Dye

Introduction

Extensive studies have been made on the use of natural products which are readily available, easily prepared, eco-friendly, less hazardous and of low cost, as indicators in acid-base titrations. These products can serve as substitutes for the synthetic compounds which are costly and harmful. These natural products can be obtained from various parts of plants like leaves, roots, fruits and stems. Example of such plant is *Hibiscus sabdariffa* known as "Roselle" (Zobo), which is rich in vitamins, natural carbohydrate, protein, tannins, gums and other antioxidants including minerals [1,2]. *Hibiscus Sabdariffa* plant has so many applications industrially and medically. Industrially, it can be used as spices for soup, sauces, wine and juice [3], infusion (herbal tea) [4], and dyes [5]. Medically, it can be used for curing allergies, high blood pressure [6], diabetes mellitus [7], for the stimulation of intestinal peristalsis [6] and for the reduction of serum cholesterol in humans [8].

In rural areas, the calyces *Hibiscus sabdariffa* can be used to improve the economic value by using it as acid-base indicator in Chemistry Laboratory in Secondary Schools. This is as a result of the presence of anthocyanin compounds in rosella flowers.

Several studies have been reported by many investigators on the effectiveness of the indicator prepared from the calyces *Hibiscus sabdariffa* for acid-base titration. The indicator changes colour over a range of hydrogen ion concentration which is expressed as the pH ranges of blue, red and colourless [9]. A study has been carried out to compare the properties of acid-base indicator of Rose, Allamanda and Hibiscus flowers [10]. Similarly, the acid-base indicator properties of dyes from *Basella alba* and *Hibiscus sabdariffa* has been determined [11]. Rosella flower has been used as alternative indicators of Blue and Red litmus [12,13]. Natural acid indicator has been isolated from the flower sap of Rosella [14,15].

Anthocyanin has been extracted from the corolla of Roselle and used as acid-base indicator. In the study, the properties of Hibiscus rosella indicator with phenolphthalein and methyl orange indicators were compared. The performance of the natural indicator was found to be similar to that of methyl orange as the results showed that the Roselle's corolla indicator gave red colour in acidic solution and green in basic solution [16]. In another study, it was found that a solution of *Hibiscus sabdariffa* crude anthocyanidins can be employed as the end-point indicator in complexometric and weak acid-weak base titrations because the end results were identical to those reproduced with standard end-point indicators, erichrome black T and phenolphthalein [17].

The objective of this research was to extract the dye (acid-base indicator) from *calyces Hibiscus sabdariffa* and to determine the acid-base indicator properties of the dye extracted from the calyces *Hibiscus sabdariffa*, for the determination of the molar concentration (standardization) of the acid and the mass concentration of the acid.

Materials and Methods

Collection of plant Materials

The calyces *Hibiscus sabdariffa* was purchased at Ekeonunwa Market, Owerri, Imo State, Nigeria, in the month of October, 2019.

Reagents and Apparatus

The reagents used were Oxalic acid ($H_2C_2O_4$), Sodium hydroxide (NaOH), Sodium carbonate (Na_2CO_3), Disodium borate ($B_4Na_2O_7$), Hydrochloric acid (HCl) and they were obtained from Mr Nti's laboratory, a Technologist in School of Agriculture and Agricultural Technology, Federal University of Technology Owerri, Nigeria. The reagents were of analytical standard.

The apparatus used were burette, conical flask, volumetric flask, beaker, funnel, automatic pipette, dropper, distillation apparatus, auto digital pH meter, heating mantle, oven, laboratory meter, electronic weighing balance, retort stand with clamp, wash bottle, spatula, stirrer, soxhlet extractor, desiccators, mechanical blender.

Preparation and Extraction of the Indicator from the calyces *Hibiscus Sabdariffa*

The flowers were washed with distilled water, dried in hot air oven at 40°C and ground into powder with mechanical blender. About 1.5 kg of the calyces *Hibiscus sabdariffa* was weighed and treated with two litres of ethanol at 70°C in a Soxhlet extractor for 72 hrs. The extract was filtered and concentrated in vacuum and finally dried in a desiccator to remove residual

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water which yielded 200 g of the crude extract.

For the hot extract, 40 g of the powder was weighed using an electronic weighing machine. This was dissolved in 80 ml of ethanol which boiled for 30 mins. It was extracted and filtered to obtain the dye solution which was concentrated to produce acid-base indicator.

For the cold extraction, the ground calyces *Hibiscus sabdariffa* was dissolved in 60 ml of cold ethanol and left overnight, the solution was later extracted, filtered and concentrated by heating to produce an indicator for acid-base titration (Table 1 & 2).

The calyces *Hibiscus sabdariffa* extract was respectively added to 0.03 M of the bases which brought about the changes in colour of the mixture (end point). At these end points, the volumes of the acids used were recorded.

Standardization of the Acid used

The mean volumes of the acids were used to determine the molar concentration and mass concentration of all the acids used in this research work. Equation 1 was used in the determination of the molar concentration

$$\frac{C_A V_A}{C_B V_B} = \frac{n_a}{n_b} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots Equation\ 1$$

where;

C_A = Concentration of Acid

C_B = Concentration of Base

V_A = Volume of Acids,

V_B = Volume of Base

n_a = number of moles of acid

n_b = number of moles of bases.

Mass Concentration = Molar Concentration × Molar mass

Results and Discussion

The mean volume of all the acids used in the experiments was shown in Table 3-10. The indicators, colour changes, molar and mass concentration of the acids used are shown in the below following tables.

Table 1. Titration of Acid/Base with the pH.

Titration	Acid /Base	Rapid change of pH
Strong acid/Strong base	HCl/NaOH	3.4 to 6.4
Strong acid/Weak base	HCl/Na_2CO_3	3.4 to 5.2
Strong acid/Weak base	HCl/$B_4Na_2O_7$	3.4 to 4.5
Weak acid/Strong base	$H_2C_2O_4$/NaOH	3.4 to 9.0
Weak acid/Weak base	$H_2C_2O_4/Na_2CO_3$	3.4 to 3.82
Weak acid/Weak base	$H_2C_2O_4/B_4Na_2O_7$	3.4 to 3.7

Table 2. Colour Changes of the calyces *Hibiscus sabdariffa*.

Dye stuff	Colour of the dye	Basic	Acids
Hibiscus sabdariffa	Wine	Green	Red

Table 3. Titration of HCl/NaOH Using Hot Ethanol Extract of *Hibiscus sabdariffa*.

Burette reading	First reading (cm^3)	Second reading (cm^3)	Third reading (cm^3)
Final reading	18.50	36.90	18.40
Initial reading	0.00	18.50	0.00
Volume of acid used	18.50	18.40	18.40

From Table 3, the mean titre value is calculated using the Equation 2 as follows:

$$Mean\ titre\ value = \frac{V_{A1} + V_{A2} + V_{A3}}{3} \quad \ldots\ldots\ldots\ldots\ldots\ldots Equation\ 2$$

where;

V_{A1} = Volume of acid used in first reading

V_{A2} = Volume of acid used in second reading

V_{A3} = Volume of acid used in third reading

$$Mean\ titre\ value = \frac{18.50 + 18.40 + 18.40}{3}$$

$$= 18.43\ cm^3$$

$$= 0.0184\ dm^3$$

From Table 4, the mean titre value was calculated using Equation 2:

Table 4. Titration of HCl/NaOH using cold ethanol extract of *Hibiscus sabdariffa*.

Burette reading	First reading (cm^3)	Second reading (cm^3)	Third reading (cm^3)
Final reading	18.80	37.60	18.80
Initial reading	0.00	18.80	0.00
Volume of acid used	18.80	18.80	18.80

$$Mean\ titre\ value = \frac{18.80 + 18.80 + 18.80}{3}$$

$$= 18.80\ cm^3$$

$$= 0.0188\ dm^3$$

From Table 5, the mean titre value was calculated using Equation 2:

Table 5. Titration of $H_2C_2O_4$/NaOH using hot ethanol extract of *Hibiscus sabdariffa*.

Burette reading	First reading (cm^3)	Second reading (cm^3)	Third reading (cm^3)
Final reading	20.30	40.70	20.40
Initial reading	0.00	20.30	0.00
Volume of acid used	20.30	20.40	20.40

$$Mean\ titre\ value = \frac{20.30 + 20.40 + 20.40}{3}$$

$$= 20.37\ cm^3$$

$$= 0.0204\ dm^3$$

From Table 6, the mean titre value was calculated using Equation 2:

Table 6. Titration of $H_2C_2O_4$/NaOH using cold ethanol extract of *Hibiscus sabdariffa*.

Burette reading	First reading (cm^3)	Second reading (cm^3)	Third reading (cm^3)
Final reading	20.50	41.00	20.50
Initial reading	0.00	20.50	0.00
Volume of acid used	20.50	20.50	20.50

$$Mean\ titre\ value = \frac{20.50 + 20.50 + 20.50}{3}$$

$$= 20.50\ cm^3$$

$$= 0.0205\ dm^3$$

From Table 7, the mean titre value was calculated using Equation 2:

$$Mean\ titre\ value = \frac{19.70 + 19.90 + 19.60}{3}$$

$$= 19.73\ cm^3$$

$$= 0.0197\ dm^3$$

Table 7. Titration of $H_2C_2O_4$/NaOH using hot ethanol extract of Hibiscus *sabdariffa*.

Burette reading	First reading (cm^3)	Second reading (cm^3)	Third reading (cm^3)
Final reading	19.70	39.60	19.60
Initial reading	0.00	19.70	0.00
Volume of acid used	19.70	19.90	19.60

Table 8. Titration of HCl/$B_4Na_2O_7$ using cold ethanol extract of *Hibiscus sabdariffa*.

Burette reading	First reading (cm^3)	Second reading (cm^3)	Third reading (cm^3)
Final reading	19.60	39.10	19.50
Initial reading	0.00	19.60	0.00
Volume of acid used	19.60	19.50	19.50

Table 9. Titration of HCl/Na_2CO_3 using hot ethanol extract of *Hibiscus sabdariffa*.

Burette reading	First reading (cm^3)	Second reading (cm^3)	Third reading (cm^3)
Final reading	20.00	40.00	20.20
Initial reading	0.00	20.00	0.00
Volume of acid used	20.00	20.00	20.20

Table 10. Titration of HCl/Na_2CO_3 using cold ethanol extract of *Hibiscus sabdariffa*.

Burette reading	First reading (cm^3)	Second reading (cm^3)	Third reading (cm^3)
Final reading	20.100	40.300	20.100
Initial reading	0.00	20.10	0.00
Volume of acid used	20.10	20.20	20.10

Table 11. Determination of Molar and Mass Concentrations.

Acid/0.03 M of Bases	Indicator	End-point/Colour change	Molar Concentration (mol/dm^3)	Volume of Base, V_B	Volume of Acid, V_A	Number of moles of Acid, n_A	Number of moles of Base, n_B	Molar Concentration (mol/dm^3) C_A	Mass Concentration (g/dm^3)
HCl/NaOH	Hot Ethanol Extract	Wine to greenish yellow	0.03	0.02	0.0184	1	1	0.0326	1.1899
	Cold Ethanol Extract	Red to light green	0.03	0.02	0.0188	1	1	0.0319	1.1644
$H_2C_2O_4$ /NaOH	Hot Ethanol Extract	Wine to colourless	0.03	0.02	0.0204	1	2	0.0147	1.3230
	Cold Ethanol Extract	Red to colourless	0.03	0.02	0.0205	1	2	0.0146	1.3140
HCl/ $B_4Na_2O_7$	Hot Ethanol Extract	Wine to pink	0.03	0.02	0.0197	2	1	0.0609	4.4457
	Cold Ethanol Extract	Light pink to colourless	0.03	0.02	0.0195	2	1	0.0615	4.4895
HCl/Na_2CO_3	Hot Ethanol Extract	Green to light green	0.03	0.02	0.0200	2	1	0.0600	4.3800
	Cold Ethanol Extract	Green to brown	0.03	0.02	0.0201	2	1	0.0597	4.3581

From Table 8, the mean titre value was calculated using Equation 2:

$$Mean\ titre\ value = \frac{19.60 + 19.50 + 19.50}{3}$$

$$= 19.53\ cm^3$$

$$= 0.0195\ dm^3$$

$$Mean\ titre\ value = \frac{20.00 + 20.00 + 20.00}{3}$$ g Equation 2:

$$= 20.00\ cm^3$$

$$= 0.0200\ dm^3$$

From Table 10, the mean titre value was calculated using Equation 2:

$$Mean\ titre\ value = \frac{20.10 + 20.20 + 20.10}{3}$$

$$= 20.13\ cm^3$$

$$= 0.0201\ dm^3$$

From the tables above, there was appearance of the sharp end point in the titrimetric analysis, these could be as a result of the presence of flavonoids and is in agreement with [10]. Between the two methods of extraction used, hot extraction yielded better because more dye was extracted having a wine colour. From the preliminary test carried out, *Hibiscus sabdariffa* extract displayed red colour in acid and green in alkaline. The colours were also observed in [11]. The mean values of the volume of acids used in the work were calculated, used in the standardization of acids and determination of mass concentration of the acids.

Conclusions

The use of Hibiscus *sabdariffa* as indicator in acid-base titration cannot be over emphasized in acid-base titration. The ethanol extract showed wine colour in acid solution, green in basic solution and it was employed for the standardization of all the acids used and for the determination of the mass concentration.

References

1. Adegunloye BJ, Omoniyi Owolabi, Ajabonna OP. "Mechanism of Blood Pressure Lowering Effects of the Calyx Extract of *Hibiscus sabdariffa* in Rats." *Journal of Science* (1996): 235-238.
2. Ahmed, Fatima Abdallah Mohammed, Nuha Mohammed Elhasan Satti, and Sharaf Eldin Hussain Eltahir. "A comparative study on some major constituents of karkade (*Hibiscus sabdariffa* L.–roselle plant)." *International Journal of Life science and Pharma Research* 9 (2019): 1-12.
3. Yadong Q, Kit LC, Fatemah M, Mila B, et al. (2005). Biological Characteristics, Nutritional and Medicinal Value of Roselle, *Hibiscus sabdariffa*. *Circular-Urban Forestry Natural Resources and Environment*, No 604. www.suagcentre.com.
4. Obidoa Onyechi, Parker Elijah Joshua, John Egemole, and Ikeyi Adachukwu. "Phyochemical Analysis of Aqueous Flower Extract of Hibiscus sadariffa (Zobo Flower)." *Research Journal of Pharmacognosy and Phytochemistry* 3 (2011): 169-173.
5. Abubakar S, Usman Etim, Nnadi, and Alaku. "Application of organic dyes from roselle calyx (*Hibiscus sabdariffa* Linn) for mycological staining." *Indian J Innovations Dev* 1 (2012): 687-690.
6. Perry "Medical plants of East and South East Asia." MIT Press, Cambridge (1980): 312-322.
7. Rosemary, Rosidah, and Ginda Haro. "Antidiabetic effect of roselle calyces extract (*Hibiscus sabdariffa* L.) in streptozotocin induced mice." *Int J PharmTech Research* 6 (2014): 1703-1711.
8. Lin Tzu-Li, Hui-Hsuan Lin, Chang-Che Chen, Ming-Cheng Lin, et al. "*Hibiscus sabdariffa* extract reduces serum cholesterol in men and women." *Nutrition research* 27 (2007): 140-145.
9. Pimpodkar Surve, and Bhise. "Use of Bixa orellana fruit extract as a natural indicator in acid base titration." *Int J Pharm Res Sch* 3 (2014): 156-159.
10. Okoduwa, Stanley IR, Lovina O Mbora, Matthew E Adu, et al. "Comparative analysis of the properties of acid-base indicator of Rose (*Rosa setigera*), Allamanda (*Allamanda cathartica*), and Hibiscus (*Hibiscus rosa-sinensis*) flowers." *Biochemistry research international* (2015).
11. Izonfuo, Fekarurhobo, Obomanu, and Daworiye. "Acid-base indicator properties of dyes from local plants I: dyes from Basella alba (Indian spinach) and *Hibiscus sabdariffa* (Zobo)." *Journal of Applied Sciences and Environmental Management* 10 (2006): 5-8.
12. Nuryanti Siti and Dwi Juli Puspitasari. "Rosella (*Hibiscus sabdariffa*) Flowers as Alternative Indicators of Blue and Red Litmus." *Oriental Journal of Chemistry* 35 (2019): 476-480.
13. Siti N. "Paper Indicator of Wora-wari Flower (Hibiscus rosa-sinensis L)." Proceeding of 3rd International Conference on Research, Implementation and Education of Mathematics and Science (2016): 95-98.
14. Ekandem JE, and Owor AU. "Local flower extracts as simple acid-base indicators." *J Chem Soc Nig* 22 (1997): 98-103.
15. Poonam Gupta, Jain Pushpa, and Jain PK. "Isolation of natural acid base indicator from the flower sap of Hibiscus rosa sinensis." *Journal of Chemical and Pharmaceutical Research* 4 (2012): 4957-4960.
16. Nuryanti Siti, Sabirin Matsjeh, Chairil Anwar, Tri Joko Raharjo, et al. "Corolla of roselle (*Hibiscus sabdariffa* L.) as acid-base indicator." *European journal of chemistry* 4 (2013): 20-24.
17. Okonkwo and Tochukwu Josiah Nnaemeka. "*Hibiscus sabdariffa* anthocyanins: a potential two-colour end-point indicator in acid-base and complexometric titrations." *Int J Pharm Sci Rev Res* 4 (2010): 123-8.

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