Senna FLOWER EXTRACT AS AN INDICATOR FOR ACID-BASE TITRATION

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ABSTRACT

Senna (Khi-Lek in Thai) is a plant that produces yellow flowers, and which grows naturally throughout Thailand. This study investigated the use in the acid-base titration of extracts from the flowers of *Senna siamea* (Lam.) H.S. Irwin & Barneby, *Senna garrettiana* (Craib) H.S. Irwin & Barneby, and *Senna surattensis* (Burm.f.) H.S. Irwin & Barneby. The dried flower extracts were added to buffer solutions, HCl/NaOH solutions, and CH₃COOH/NaOH solutions, and any color change was observed. All three extracts caused the solutions to become darker brown as the pH increased. *Senna surattensis* Burm. f. the extract produced the most easily observed change. This extract was therefore shown to be an effective indicator in acid-base titration of 0.1 M HCl and 0.5 M NaOH. The solution changed visibly from colorless to brown. Its effectiveness compared favorably with that of phenolphthalein, a standard laboratory indicator. *Senna* extract is particularly appropriate for use in chemistry demonstrations at Thai schools as it is widely available, economical, easy to prepare, safe in use, and environmentally friendly.

Keywords: *Senna surattensis* Burm. f., *Senna garrettiana* Craib, *Senna siamea* Lam., Indicator, Acid-base titration.
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INTRODUCTION

An indicator is a halochromic chemical compound that, when added in small amounts to a solution, changes color at a specific pH or in a way that measures acidity, alkalinity, or the progression of a reaction.¹ It is used in an acid-base titration, a basic method of analyzing chemical content in which an acid and base at a known concentration and standard solution (titrant) are reacted in an Erlenmeyer flask with a solution of unknown concentration (titrant). Equivalence points are identified when the indicator changes color. The endpoint or color change selected should be close to the equivalence point of the acid-base reaction.²⁻⁴ Most chemical laboratories use synthetic acid-base indicators. However, natural extracts are safer, cheaper, and easier to source, making them a desirable substitute.^{5,6} Natural flavonoids such as anthocyanins and flavanones have been demonstrated to act as indicators.⁷⁻⁹ The current study focused on extracts from the flowers of three *Senna* species, as these contain flavonoids and therefore were proposed to have the chemical properties of an indicator

Senna siamea (Lam.) H.S. Irwin & Barneby is a perennial of the Fabaceae family that reaches a height of 8-15 m. The trunk is often bent, with gray to brown-black bark that has shallow furrows running along its length. The leaves are dark green and made up of alternately arranged feathers. The plant bifurcates into a bouquet of branches tipped with yellow flowers.^{10,11} Its extracts are used extensively in traditional Thai medicine to treat constipation and to promote blood, bile, and appetite. The phytochemical constituents of *S. siamea* Lam. include polyphenols,¹² sterols,¹³ chromones,¹⁴ alkaloids,¹⁵ and flavonoids.¹⁶⁻¹⁸

Senna garrettiana (Craib) H.S. Irwin & Barneby (Fabaceae) is another perennial that reaches a height of 10 m. The trunk is straight and its thick bark is dark brown to black. The leaves take the form of an elongated oval with a spear-like tip and the flowers are yellow to golden in color. The skin of the pod is smooth and

completely hairless.^{19,20} Phytochemical studies of *S. garrettiana* Craib have identified a range of compounds including chrysophanol, cassialoin, cassigarols A-G, chrysophanic acid, chrysophanol dianthrone, quercetin, piceatannol, piceatanol, protocatechuic aldehyde, scirpusin B, and betulic acid. Extracts have been reported to be cytotoxic against cancer cells and embryos, to stimulate the uterus, and to prevent the excretion of gastric juice.²¹

Senna surattensis (Burm. f.) H.S. Irwin & Barneby (Fabaceae) is a small- to a medium-sized shrub that grows up to 7 m in height. The smooth leaves comprise a single feather layer, and sparse hair is arranged alternately between the leaves and the belly. Flowers appear as a bouquet of elbows near the tips of the branches. The petals are oval and yellowish green in color. The pods are flat and smooth with a shiny seed surface. *S. surattensis* Burm. f. is used as a herbal remedy against fever and hiccups.¹⁹ Aqueous extracts of this plant have been found to contain flavonoids, alkaloids, steroids, and amino acids.²²

Little information is available on the chemical content of *Senna* flowers, but they are believed to contain flavonoids.^{23,24} This suggests potential applications as an indicator for acid-base titration, though a literature search turned up no reports of the extracts being used in this way. In this study, we investigated the color change in *Senna* flower extracts when exposed to solutions of specific pH. We tested them as an indicator for acid-base titration and compared their performance with that of a synthetic indicator. If an indicator derived from *Senna* flowers can replace synthetic indicators it will find a special role as a teaching aid in Thai schools, as *Senna* is widely available, cheap, convenient, and safe for the user and the environment.

EXPERIMENTAL

Flower extraction

Petals of *S. siamea* Lam., *S. garrettiana* Craib, and *S. surattensis* Burm. f. was collected from Suphanburi province, Chanthaburi province, and Bangkok province, respectively. After drying at 60 °C for two hours, extraction was performed at 100 °C for one hour using 6 g of dried leaf and 200 mL of deionized water. After cooling to room temperature, the aqueous extracts were passed through white filter cloth and Whatman number 1 filter paper. They were placed in a vacuum rotary evaporator at 60 °C to prepare 5 % (w/w) dried extract in water.

Color Change when placed in pH Solution

Five drops of 5%(w/w) extract was added to three solutions: buffer (pH 1-13), hard acid–hard base (pH 1-14, prepared from hydrochloric acid (HCl) and sodium hydroxide (NaOH)), and soft acid–hard base (pH 1-14, prepared from acetic acid (CH₃COOH) and NaOH). The buffer solutions used were mixtures of the following: HCl/potassium chloride (KCl) at pH 1 and 2, HCl/potassium hydrogen phthalate (KHP) at pH 3 and 4, KHP/NaOH at pH 5, potassium dihydrogen phosphate (KH₂PO₄)/NaOH at pH 6 to 8, HCl/sodium tetraborate decahydrate (Na₂B₄O₇·10H₂O) at pH 9, HCl/sodium bicarbonate (NaHCO₃) at pH 10 and 11, and KCl/NaOH at pH 12 and 13.²⁵⁻²⁷

Titration

Those extracts that exhibited the strongest color change were chosen as the indicator for strong acid–strong base titration (1.0 HCl titrated with 0.5 NaOH and 1.0 M NaOH) and weak acid–strong base titration (1.0 M CH₃COOH titrated with 0.5 NaOH and 1.0 M NaOH). Five drops of extract were mixed with 15 mL of acid solution in a 50 mL beaker. Titration with KHP solution was used to find the concentration of the base. The experiments were repeated five times and the average base volumes and standard deviations were compared with results obtained using phenolphthalein, a standard laboratory indicator. A pH meter was used to measure the endpoint pH.

RESULTS AND DISCUSSION

Color Change of 5%(w/w) Flower Extract when exposed to Solutions of Different pH

Buffer solutions of higher pH changed in color to darker brown when *Senna* extracts were added. The change was most apparent when *S. surattensis* Burm. f. was added to solutions with pH 10-13 (Fig.-1).

Senna extracts were added to pH 1-14 mixtures of HCl and NaOH. The addition of *S. siamea* Lam. and *S. garrettiana* Craib extract induced a color gradient from light brown to darker brown, reflecting the change

in pH. When *S. surattensis* Burm. f. extract was added, the solution was very light brown in the pH 1-9 range and dark brown in the pH 10-14 range (Fig.-2).

When added to CH_3COOH -NaOH solutions of pH 1-14, *S. siamea* Lam. and *S. garrettiana* Craib extracts induced a gradient from light brown to darker brown as the pH increased. *S. surattensis* Burm. f. extract produced a light brown color at pH 1-10 and dark brown color at pH 11-14 (Fig.-3).

The buffer solutions changed to darker brown as the pH increased, suggesting that the extracts may be used in strong acid–strong base titration (for example HCl-NaOH titration) or weak acid–strong base titration (for example CH_3COOH -NaOH titration). The color change was produced by flavonoids in the *Senna* extracts^{7, 8, 28} which are brown when in base solution. *S. surattensis* Burm. f. extract produced the clearest color contrast when added to acid and base solutions. The color change appeared at pH 10-13 for buffer solutions, at pH 10-14 for HCl/NaOH, and pH 11-14 for CH_3COOH /NaOH.

Fig.-1: Color Change in pH 1-13 Buffer Solutions with the Addition of Extract from (a) *S. siamea* Lam., (b) *S. garrettiana* Craib, and (c) *S. surattensis* Burm.f.

Fig.-2: Color Gradient of pH 1-14 HCl-NaOH Solutions after Addition of (a) *S. siamea* Lam., (b) *S. garrettiana* Craib, and (c) *S. surattensis* Burm.f.

Fig.-3: Color Gradient of pH 1-14 CH₃COOH-NaOH Solutions after Addition of (a) *S. siamea* Lam., (b) *S. garrettiana* Craib (c), and *S. surattensis* Burm.f.

Titration

S. surattensis Burm. f. extract was tested as a titration indicator and its performance was compared with that of phenolphthalein, a standard laboratory indicator that exhibits a clear change in color when added to acid and base solutions. Table-1 shows the results. In HCl-NaOH titration with extract, the HCl solution was colorless before titration, becoming brown at the endpoint of titration with 0.5 M NaOH and light brown at the endpoint of titration with 1.0 M NaOH (Fig.-4).

Table-1: Acid-Base Titration using Phenolphthalein and *S. surattensis* Burm. f. Extract as an Indicator

Titrand	Indicator	Mean values of 0.5 M NaOH $\pm$ S.D. (mL) ^a (Color change)	Mean values of 1.0 M NaOH $\pm$ S.D. (mL) ^a (Color change)	pH
1.0 M HCl	Phenolphthalein	29.19 $\pm$ 0.06 (Colorless to Pink)	14.56 $\pm$ 0.11 (Colorless to Pink)	8.22 -10.05
	<i>S. surattensis</i> Burm. f. extract	29.17 $\pm$ 0.08 (Colorless to Brown)	15.00 $\pm$ 0.52 (Colorless to Light brown)	9.34 -12.14
1.0 M CH ₃ COOH	Phenolphthalein	29.29 $\pm$ 0.11 (Colorless to Pink)	14.64 $\pm$ 0.11 (Colorless to Pink)	8.22 -10.01
	<i>S. surattensis</i> Burm. f. extract	29.72 $\pm$ 0.15 (Colorless to Brown)	14.80 $\pm$ 0.08 (Colorless to Light brown)	11.14 -11.76

^aAll values are means $\pm$ SDs from five replications.

Fig.-4: (a) HCl with Extract before Titration, (b) HCl at the endpoint of titration with 0.5 M NaOH, (c) HCl at the endpoint of titration with 1.0 M NaOH

In the titration of CH_3COOH and NaOH , the initial solution of CH_3COOH and *S. surattensis* Burm. f. extract was colorless. When titrated with 0.5 M NaOH , the solution became brown by the endpoint. When titrated with 1.0 M NaOH , the solution became light brown by the endpoint (Fig.-5).

Fig.-5: (a) CH_3COOH solution with extract before titration, (b) CH_3COOH solution at the endpoint of titration with 0.5 M NaOH , (c) CH_3COOH solution at the endpoint of titration with 1.0 M NaOH

The concentration of NaOH was reflected in the color intensity of the solution, which was darker brown when titrated with 0.5 M NaOH than with 1.0 M NaOH . A color gradient was apparent at the endpoint, and the estimate of pH was close to that given by phenolphthalein. Titration with 0.5 M NaOH produced a positive error, as the light brown color was difficult to distinguish. The base volumes and color changes were the same when HCl and CH_3COOH were used as the titrant. The endpoints were at pH 9.34-12.14 when using HCl and at pH 11.14-11.76 when using CH_3COOH . The use of HCl as titrant produced a wide range of endpoints, whereas the use of CH_3COOH produced a narrow pH range and a strong base period. *S. surattensis* Burm. f. extract was shown to be a practical indicator for use in strong acid-strong base titration, optimally at 5%(w/w) for titration of 0.1 M HCl and 0.5 M NaOH . As it can be derived from naturally-occurring plants and extracted using water as a solvent, it is cheap and easy to prepare, safe to use, and environmentally neutral.

CONCLUSION

This research investigated the color change in extracts from *S. siamea* Lam., *S. garrettiana* Craib, and *S. surattensis* Burm. f. when added to solutions of different pH. Their performance as indicators in titration was compared with that of a standard laboratory indicator. All three extracts induced a change in color to darker brown as the pH of the solution was increased. Extract from *S. surattensis* Burm. f. produced the most easily observed color change. When used as an indicator in HCl - NaOH titration, the solution changed from colorless to brown at pH values of 9.34-12.14. In CH_3COOH - NaOH titration, the change occurred at pH 11.14-11.76. When the base concentration was stronger, the brown color of the solution was weaker. The performance of *S. surattensis* Burm. f. extract was closest to that of phenolphthalein when used in the titration of 1.0 M HCl and 0.5 M NaOH . *Senna* is readily available throughout Thailand. By simple extraction with water, the flowers yield an indicator with practical applications in acid-base titration. It is particularly appropriate for school chemistry demonstrations as it is economical, easy to prepare, safe in use, and environmentally friendly.

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REFERENCES

1. Encyclopedia.com, Available from: <https://www.encyclopedia.com/environment/encyclopedias-almanacs-transcripts-and-maps/indicator>.
2. D. Harvey, Modern Analytical Chemistry, McGraw-Hill, Boston, p. 273(2000).

3. D.C. Harris, Quantitative Chemical Analysis, Acid-Base Titrations, Craig Bleyer, New York, p. 199 (2007).
4. K.W. Whitten, R.E. Davis, M.L. Peck and G.G. Stanley, Chemistry, Cengage Learning, Belmont CA, p. 379(2010).
5. N.S. Aminah, A.P. Wardana, A.N. Kristanti, B.V. Safitri and M. Rosa, *Rasayan Journal of Chemistry*, **12(4)**, 2116(2019), DOI:10.31788/RJC.2019.1245498
6. T. Tukiran and A.P. Wardana, *Rasayan Journal of Chemistry*, **11(2)**, 773(2018), DOI:10.7324/RJC.2018.1121844
7. S. Singh, S.B. Bothara and S. Singh, *Pharmacognosy Journal*, **3(19)**, 35(2011), DOI:10.5530/pj.2011.19.7
8. J.M. Bueno, P. Sáez-Plaza, F. Ramos-Escudero, A.M. Jiménez, R. Fett and A.G. Asuero, *Critical Reviews in Analytical Chemistry*, **42(2)**, 126(2012), DOI:10.1080/10408347.2011.632314
9. M. Zulfajri and Muttakin, *Rasayan Journal of Chemistry*, **11(1)**, 135(2018), DOI:10.7324/RJC.2018.1111983
10. Medthai, Available from: <https://medthai.com/พืชหลัก/>
11. M. Hongsirinirachorn, S. Threeprasertsuk, A. Chutaputti, *Journal of the Medical Association of Thailand*, **86(6)**, 484(2003).
12. G. Kaur, M.S. Alam, Z. Jabbar, K. Javed and M. Athar, *Journal of Ethnopharmacology*, **108(3)**, 340(2006), DOI:10.1016/j.jep.2006.05.021
13. P. Ghosh, S. Thakur, T. Itoh and T. Matsumoto, *Indian Journal of Chemistry Section B: Organic Chemistry Including Medicinal Chemistry*, **21**, 796(1982).
14. B.Z. Ahn, U. Degen, C. Lienjayetz, P. Pachaly and F. Zymalkowski, *Archiv der Pharmazie*, **311(7)**, 569(1978), DOI:10.1002/ardp.19783110703
15. S. Oshimi, J. Deguchi, Y. Hirasawa, W. Ekasari, A. Widyawaruyanti, T.S. Wahyuni, N.C. Zaini, O. Shiota and H. Morita, *Journal of Natural Products*, **72(10)**, 1899(2009), DOI:10.1021/np9004213
16. O. Kaisoon, S. Siriamornpun, N. Weerapreeyakul and N. Meeso, *Journal of Functional Foods*, **3(2)**, 88(2011), DOI:10.1016/j.jff.2011.03.002
17. A. Bakar, M. Dauda and A. Hassan, *Bayero Journal of Pure and Applied Sciences*, **2(1)**, 139(2009), DOI:10.4314/bajopas.v2i1.58528
18. M. Kamagate, C. Koffi, N.M. Kouamé, A. Akoubet, N.A.R. Yao and H.M. Die-Kakou, *The Journal of Phytopharmacology*, **3(1)**, 57(2014).
19. N. Rueangrangsi and T. Mangkhlahup, Thai Herbal Books, Be Healthy Publisher, Bongkok, p. 141 (2003).
20. P. Subcharoen, Herbs in National Park of Central Region, Thai Traditional Medicine Textbook, Nonthaburi, p. 145 (2006).
21. Thongthailand, Available from: <http://www.thongthailand.com/index.php?lite=article&qid=307467>
22. A.E.-M.M.R. Afify and H.M.M. Hassan, *Asian Pacific Journal of Tropical Biomedicine*, **6(9)**, 771 (2016), DOI:10.1016/j.apjtb.2016.07.006
23. T. Iwashina, *Natural Product Communications*, **10(3)**, 529(2015), DOI:10.1177/1934578X1501000335
24. J.B. Harborne, The Flavonoids: Advances in Research since 1980, Springer Science+Business Media, Dordrecht, p. 525(1988).
25. R.A. Robinson, R.H. Stokes, The Measurement and Interpretation of Conductance, Chemical Potential, and Diffusion in Solutions of Simple Electrolytes, Electrolyte Solutions, Butterworths, London, (1968).
26. D.D. Lloyd, AnalChem Resources, Available from: <http://delloyd.50megs.com/moreinfo/buffers2.html>.
27. N. Pattarapongdilok, P. Malichim, N. Simmee and J. Sichaem, *International Journal of Chemical Engineering and Applications*, **10(1)**, 24(2019), DOI:10.18178/ijcea.2019.10.1.734
28. C. Paul, D.D. Chakraborty, A. Ghosh and P. Chakraborty, *Journal of Applied Pharmaceutical Science*, **4(9)**, 120(2014), DOI:10.7324/JAPS.2014.40921

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