

ORIGINAL RESEARCH PAPER

Areca catechu seed extract as improvised acid-base indicator in titrimetric Analysis: An environmental benign approach

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ABSTRACT

At present, the synthetic compounds are wide choice as indicators in various acid-base titrations. Due to strict environmental regulations, search for eco-friendly compounds as an effective indicator of various acid-base titrations was started. The current vocation highlights the exploit of Areca catechu seed extract as an efficacious indicator for various acid-base titrations and to determine their K_a values. The Areca catechu seed is easily available and easy to extract. The extraction performed using the Soxhlet extraction apparatus. UV-Vis spectroscopy, FT-IR spectroscopy, XRD, physical properties (density, viscosity, surface tension, and refractive index) and qualitative phytochemical screening was performed for the proper identification of the Areca catechu seed extract. The Areca catechu seed extract exhibits sharp color change at the endpoint during the various acid-base titrations. The specific contrast between their colors in both the acid and alkali media made species present in the Areca catechu seed extract suitable for the eco-friendly indicator for four acid-base titrations. The endpoint obtained by the extract of Areca catechu seed coincides with the endpoint obtained by the standard synthetic indicator. Areca catechu seed is found to be economical, useful, accurate, simple, and eco-friendly.

Keywords: *Areca catechu seed; Indicator; Acid-base titration; Density; Eco-friendly; Refractive index*

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INTRODUCTION

Titration is a fundamental technique used for the quantitative analysis of the unknown concentration of the substance using the standard known concentration solution. The determination of endpoint is very difficult in the case of acid-base titration because the reaction between the acid and base yields water and salt which are colorless. Hence, an indicator is required to determine the endpoint in acid-base titration. Indicators are the chemical substance, that alters the color according to the hydrogen ion concentration in the solution [1-3]. The indicators employed in the acid-base titration are usually commercial (synthetic) inhibitors. Commercial inhibitors are generally toxic, availability problems and expensive on the

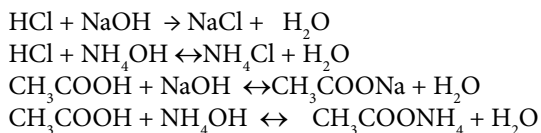
living creatures and also posses' environmental pollution [4-7]. A nation becomes aware of ecological issues. Hence, chemical researchers focused on the investigation of natural indicators, which are regarded as a symbol for green chemistry and a very good substitute for the synthetic inhibitors for various acid-base titrations. This substitute will be easier, more available, cheaper, and environmentally friendly. Several plant extract species express unique color characters in various pH. Pigments and dyes present in the plant extract species are colored and show variations in the acid and base systems. This nature can be employed to use as an eco-friendly inhibitor for various acid-base titrations. Different inorganic and organic species, mainly responsible for the color property of the

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green extract such as carotene, flavonoids, acylated flavonoids, glucosylated acylated anthocyanin, imines, polymethines, naphthoquinones, anthraquinonoids, diarylmethanes, dihydropyrans, and indigoids. All these compounds are water-soluble. Further, the plant extract species found to be significant in current laboratories when automatic titrators unable to titrate certain reactive liquids. The rationale behind in current investigation is to tap the feasibility of green indicators in acid-base titration methods which replaces the artificial indicators. Therefore, research shifted towards cheap and eco-friendly corrosion indicators. Plant products fulfill these characteristics [8-18]. The areca nut is the seed of the Areca palm, which generally grows in the southeast, South Africa, and tropical pacific. The areca nut seed contains many color compounds in their moiety (rutin, procyanidin B1, leucocyanidin, catechin, epicatechin, and gallic acid) which show different color at different pH [19-21]. Hence, in the current investigation, the potential of Areca catechu seed extract as an indicator in acid-base titrations was assessed. The endpoint in four different acid-base titrations is screened based on the color change of the plant extract. The acid-base titrations such as strong acid-strong base, weak acid-strong base, strong acid-weak base, and weak acid-weak base were performed using the Areca catechu seed extract as indicators and accuracy compared with synthetic phenolphthalein indicator. The acid-base

titration carried out three times and average values were reported. Further, their K_a values were also reported.

The chemical reactions take place during the various acid-base titrations are shown below:



The work plan of the present investigation is depicted below:

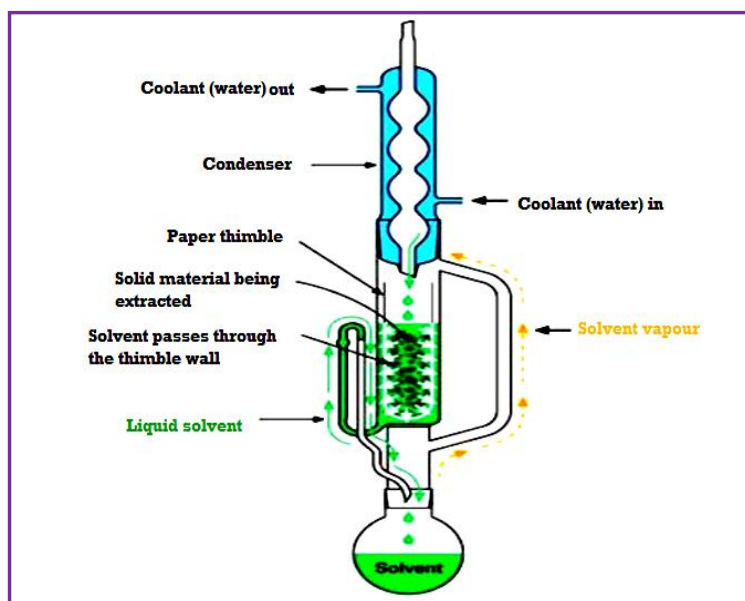
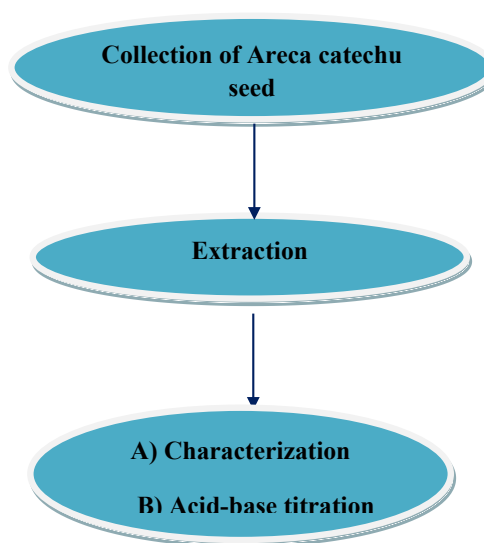


Fig. 1. Soxhlet device (schematic representation)

EXPERIMENTAL SECTION

Preparation of Areca catechu seed extract and characterization

500 g of Areca catechu seed extract was extracted with the help of Soxhlet extractor with 500 ml of double-distilled water (schematic representation shown in Fig. 1.) After the successful operation, the extract placed in the refrigerator to prevent the degradation of the sample. The presence of functional groups in the green extract was analyzed by FT-IR spectroscopy (wavenumber range 4000 to 400 cm^{-1}) and λ_{max} value was determined by using the UV-Vis spectroscopy technique. The amorphous or crystalline nature of Areca catechu seed extract was confirmed with the aid of the X-RD technique.

Materials and chemicals

For various acid-base titrations, the analytical grade of sodium hydroxide (NaOH), hydrochloric acid (HCl), ammonia (NH_3), and acetic acid (CH_3COOH) was used in the present investigation. The extract of Areca catechu seed is treated as indicator for four acid-base titration systems (0.5 M HCl (strong acid) vs 0.5 NaOH (strong base), 0.5 M HCl (strong acid) vs NH_4OH (weak base), 0.5 M CH_3COOH (weak acid) vs 0.5 M NaOH (strong base), 0.5 M CH_3COOH (weak acid) vs 0.5 M NH_4OH (weak base). 5 ml of acid along with the two drops of extract of Areca catechu seed was titrated against the respective bases. The change in the color was observed and the results are tabulated. Further, reproducibility was carefully screened by performing each titration for 3 times.

RESULTS AND DISCUSSION

Phytochemical analysis

The green chemicals responsible for the indicator property present in the Areca catechu seed extract was confirmed by the phytochemical studies. Phytochemicals played a significant role in the various acid-base titrations by using the plant extract species as a green indicator because its color and chemical structures change with the pH of the environment. The preliminary phytochemical qualitative analysis of Areca catechu seed extract shows the presence of different green chemical groups like flavonoids, alkaloids, proteins, phenols, and carbohydrates in their moiety. The results are shown in Table 1 and Fig. 2.

Physical properties

Surface tension is one of the physical properties, which gives information about the nature of the force of attraction between the two molecules in the liquid. In the present investigation, the drop count method (by using stalagmometer) employed for the determination of surface tension. Viscosity is another physical property, explores the information about how fluid resists the forces. It is the mechanical friction between the molecules in the motion and determined by using Viscometer. Density is the ratio of the mass of the substance to its volume. The density is measured by using the specific gravity bottle. Further, the refractive index (index of refraction) measures the bending of a light ray when traveling from one medium to another medium. Abbe refractometer used for the determination of the refractive index of

Table 1. Phytochemical screening results

Phytoconstituents	Indication
Test for alkaloids (Wagner's reagent): Areca catechu seed extract shows reddish-brown precipitate with Wagner's reagent (solution of iodine in potassium iodide).	Positive
Test for Flavonoids (Zn test): 5 ml of Areca catechu seed extract treated with zinc dust and concentrated hydrochloric acid, shows red color, which is an indication of the presence of Flavonoids in the extract.	Positive
Test for phenols (ferric chloride test): Areca catechu seed extract was treated with four drops of alcoholic ferric chloride solution. The formation of bluish color shows the presence of phenols.	Positive
Test for proteins (xanthoproteic test): Areca catechu seed extract was treated with a few drops of concentrated nitric acid. The formation of yellow color shows the presence of proteins.	Positive
Test for carbohydrates (benedict's reagent): The filtrate was treated with benedict's reagent and heated gently. Orange-red precipitate shows the presence of reducing sugars.	Positive

the plant extract. All the physical parameters recorded for three times and average values are recorded. Therefore, in this investigation, for proper identification of Areca catechu seed

extract, physical properties such as density (g/ml), refractive index (n_{D20}), surface tension (dynes/cm) and viscosity (centipoise) were also determined and results are shown in Table 2.

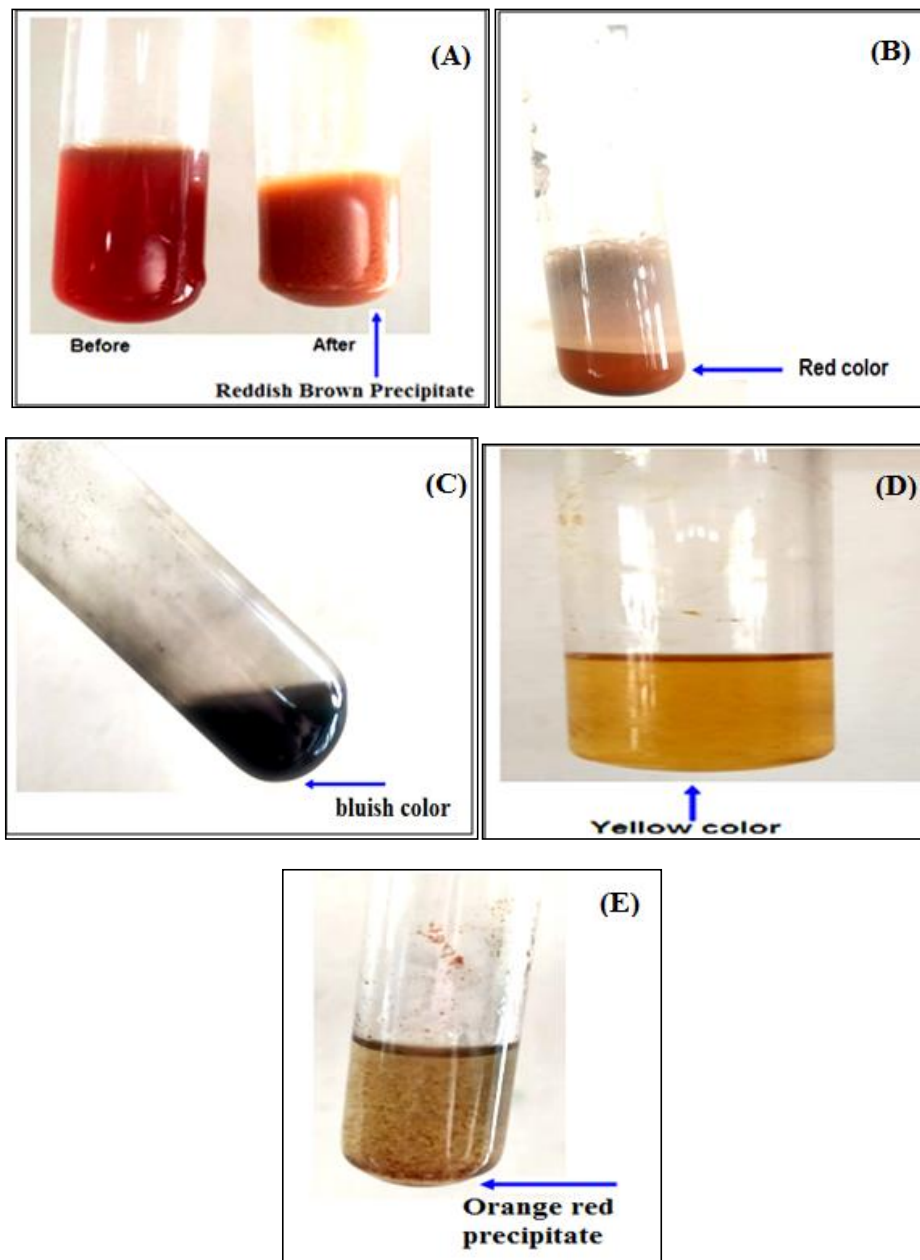


Fig. 2. Photochemical screening results, test for A) Alkaloids, B) Flavonoids, C) Phenols, D) Proteins and E) Carbohydrates

Table 2. Physical parameters of Areca catechu seed extract at ambient temperature (303 K)

Indicator	Refractive index (n_{D20})	Surface tension (dynes/cm)	Viscosity (centipoises)	Density (g/ml)
Areca catechu seed extract	1.338	83.733	0.986	1.119

UV-Vis spectroscopy

UV-Vis spectroscopy is optical absorption spectroscopy used for exploring electronic and band structure-property of plant extract. Areca catechu seed extract was centrifugated at 5000 rpm for about 30 minutes. After that, the resulting solution filtered by using the Whatman number one filter, then the sample is diluted to the 1:20 by using the double-distilled water as a solvent. The UV-Vis spectrum was recorded in the range of 200 nm to 350 nm. The results are shown in Fig. 3. The UV-Visible spectroscopy of Areca catechu seed extract shows broadband at 280 nm (assigned to $n \rightarrow \pi^*$ transitions). This absorption band is a characteristic of the flavonoids and which may be the main reason for its activity as a good natural acid-base indicator. This is following the previous report [22].

The optical band gap energy can be determined by using observed UV-Vis spectra through Tauc's plots (Fig. 4).

Further, the energy band gap can be calculated from the Tauc equation,

$$ah \nu \propto (h\nu - E_g)^n \quad [23]$$

where, ν = frequency of light, h = Planck's constant, and E_g = bandgap energy.

The energy bandgap for the four different concentrations of Areca catechu seed extract (0.1 mg/L, 0.2 mg/L, 0.3 mg/L, and 0.4 mg/L) is shown in Fig. 4. The values are in between the range of 4.72 to 4.75 eV.

Powder X-ray diffraction technique

X-ray diffraction (X-RD) is an experimental technique that gives information about the physical properties of the plant extract, chemical composition, and crystallographic structure. Hence, in the current investigation, the X-RD of Areca catechu seed extract was recorded at 40 kV in a 1.5406 \AA . The crystalline size (p) obtained by using Scherrer's equation:

$$\frac{K\lambda}{(\beta \cos\theta)} = P \quad [24]$$

Where, $k=0.9$,
 β = width at half maximum intensity, $\lambda = 1.5405 \text{ \AA}$

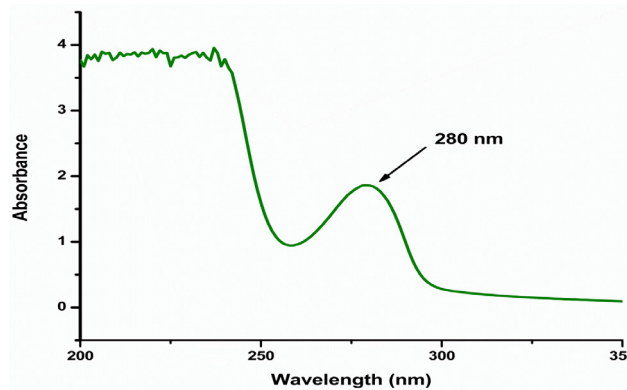


Fig. 3. UV-Vis spectrum of Areca catechu seed extract

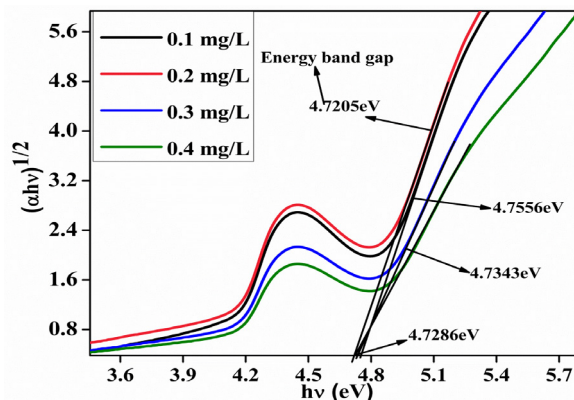


Fig. 4. Tauc plot

Interplanar spacing (d), quality factor (Q), intercrystalline separation (R) and degree of crystalline are obtained by using Scherrer's equation:

Average intercrystalline separation (R) was calculated from the below relation;

$$\frac{5\lambda}{(8\sin\theta)} = R \quad [24]$$

The results are shown in Fig. 5. The intensive peaks in the Areca catechu seed extract clearly show the crystalline nature of the plant extract. The various results such as crystalline size (p), interplanar spacing (d), quality factor (Q), intercrystalline separation (R), and degree of crystalline obtained from the X-RD are shown in Table 3.

FT-IR results

FT-IR spectroscopy is a powerful tool that can be widely employed for the determination of the functional groups present in the plant extract. The available functional groups in the Areca catechu seed extract were identified by the FT-IR spectroscopy technique (Fig. 6), which are presented in Table 4. From this table, it is clear that Areca catechu seed extract contains hydroxyl and carbonyl groups in their moieties. This group is responsible for the structural changes of the Areca catechu seed extract in hydrochloric acid and sodium hydroxide medium environment.

Indicator studies

Areca catechu seed extract exhibits different colors in the acidic and basic medium and endpoint determined for the different acid-base titrations by

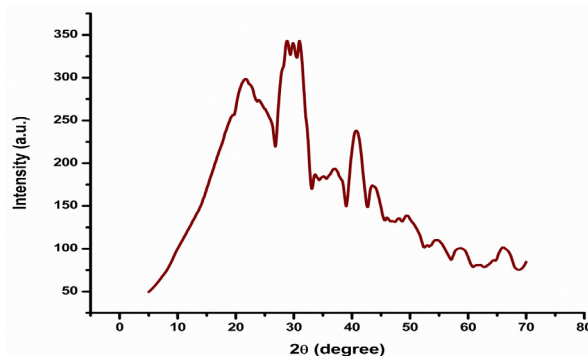


Fig. 5. XRD pattern of Areca catechu seed extract

Table 3. XRD results

2θ	θ	<P>nm	d(am)	Q(Å)	R(Å)	Degree of Crystalline
21.685	10.842	1.972	4.995	1.534	5.118	
28.839	14.419	2.003	3.773	2.031	3.866	
29.843	14.921	2.005	3.649	2.100	3.739	
30.958	15.479	2.010	3.521	2.176	3.607	
33.675	16.837	2.024	3.244	2.362	3.324	
35.123	17.561	2.032	3.114	2.461	3.191	
37.105	18.552	2.043	2.953	2.595	3.026	
40.759	20.379	2.067	2.698	2.840	2.765	
43.53	21.760	2.086	2.534	3.024	2.596	47.01492
48.075	24.037	2.121	2.307	3.322	2.363	
49.545	24.772	2.134	2.242	3.417	2.297	
54.381	24.864	1.944	11.081	0.691	11.354	
58.694	29.347	2.222	1.917	3.997	1.964	
62.063	31.031	2.261	1.822	4.204	1.867	
65.974	32.987	2.310	1.726	4.440	1.768	
69.99	34.995	-0.069	1.638	4.677	1.678	

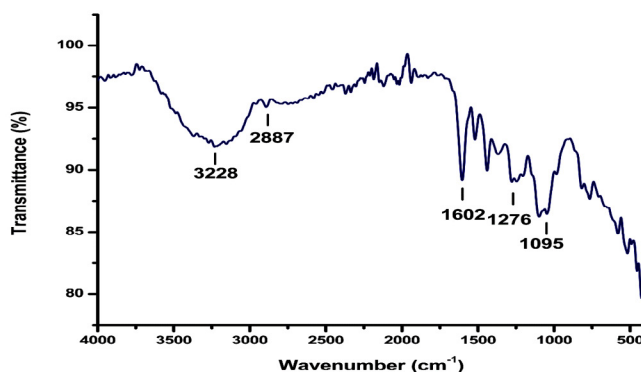


Fig. 6. FT-IR spectrum of Areca catechu seed extract

Table 4. Results of the FT-IR spectrum of Areca catechu seed extract

Bands	Groups
3228 cm ⁻¹	Monomeric hydroxyl
1602 cm ⁻¹	Carbonyl
1276 cm ⁻¹	C-H stretching vibrations
1095 cm ⁻¹	C-O
2887 cm ⁻¹	O-C=O

using the extract of Areca catechu seed is found to be similar to standard synthetic indicators employed in the laboratories. Areca catechu seed extract act as a good class of natural indicator by changing its color when it is reacted with acid and base solutions. The intense and sharp color change is due to structural changes of Areca catechu seed extract species.

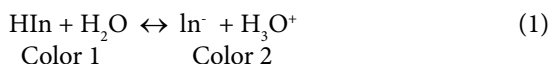
The equivalence point obtained with Areca catechu seed extract for four different systems compared with the same system with the addition of two drops of phenolphthalein indicator. The

green indicator property of an extract of Areca catechu seed is screened by using the strong acid-strong base, strong acid-weak base, weak acid-weak base, and weak acid-strong base. The results are shown in Table 5. In a strong acid-strong base system, the extract of Areca catechu seed exhibits an endpoint observation yellowish to reddish at 5 ml of titrant quantity. Whereas, the synthetic phenolphthalein showed endpoint at 4.8 ml. In the case of a strong acid-weak base system, the extract of Areca catechu seed acts as an indicator and shows the endpoint by changing the color from yellowish to reddish at 3.7 ml, for the same system, the synthetic phenolphthalein shows endpoint at 4.4 ml. The titration of the type weak acid-strong base, Areca catechu seed extract shows endpoint (from yellowish to reddish) at 5.4 ml of titrant volume. The synthetic phenolphthalein shows an endpoint at 5.1 ml. In the weak acid-weak base system, Areca catechu seed extract act as a good green indicator at 4 ml of titrant volume by changing color from

Table 5. Titrimetric analysis results

Titration	Endpoint Indicator= (Phenolphthalein indicator)	Endpoint Indicator= (Areca catechu seed extract)	Burette readings (indicator= Phenolphthalein)	Burette readings (indicator= Areca catechu seed extract)	pH range of Areca catechu seed extract	K _a	% error
0.5 M HCl (5m) vs 0.5 M NaOH	Colorless to pale pink	Yellowish to Reddish	i) 4.6 ml ii) 4.9 ml iii) 4.9 ml	i) 4.6 ml ii) 5.4 ml iii) 5.0 ml	4.70-7.30	0.009-0.0006	7.96 × 10 ⁻⁵
0.5 M CH ₃ COOH vs. 0.5 M NaOH	Colorless to pale pink	Yellowish to Reddish	i) 5.2 ml ii) 5.1ml iii) 5.2 ml	i) 5.3 ml ii) 5.5 ml iii) 5.4 ml	5.22-8.97	0.0054-0.00012	3.88 × 10 ⁻³
0.5 M CH ₃ COOH vs. 0.5 M NH ₄ OH	Colorless to pale pink	Yellowish to Reddish	i) 4.4 ml ii) 3.8 ml iii) 3.8 ml	i) 4.1 ml ii) 4.3 ml iii) 3.8 ml	5.29-8.40	0.005-0.00022	9.130 × 10 ⁻⁴
0.5 M HCl (5m) vs 0.5 M NH ₄ OH	Colorless to pale pink	Yellowish to Reddish	i) 4.9 ml ii) 4.3 ml iii) 4.0 ml	i) 3.5 ml ii) 3.9 ml iii) 3.8 ml	3.70-8.08	0.024-0.0003	4.20 × 10 ⁻⁴

yellowish to reddish. The phenolphthalein shows endpoint at 4 ml of titrant volume. The above results show that Areca catechu seed extract acts as a universal green indicator and shows a close endpoint compared with standard synthetic indicator. In all the four titrations, the extract of Areca catechu seed was found to be accurate and very useful for determining the endpoint. Areca catechu seed extract provides satisfactory results compared to the synthetic indicators with the almost same degree of accuracy. The proposed Areca catechu seed extract as an indicator is inexpensive and imparts a color change at the endpoint. Thus, from the view of green chemistry, Areca catechu seed extract can be successfully used for various routine acid-base titrations. Hence, the synthetic indicators are replaced by Areca catechu seed extract as they are cheap, easily available, precise, and accurate and prepared by a simple method. The natural indicators are the weak acid or weak base which shows the various colors at different pH. The natural indicator equilibrium reaction is shown in the below reaction:



Many indicators are present as HIn in a strong acid system and show the respective color of HIn. In a strong basic system, many indicators present as In⁻ and shows the respective color of In⁻.

The equilibrium constant can be written as:

$$K_a = \frac{[\text{In}^-][\text{H}^+]}{[\text{HIn}]}$$

At equilibrium (half equivalence point), the concentration of In⁻ and HIn are same. Hence, the above equation becomes;

$$K_a = \text{H}^+$$

Take -log on both the sides, the above equation becomes,

$$-\log (K_a) = -\log (\text{H}^+)$$

$$\text{pK}_a = \text{pH}$$

$$\text{Hence, } K_a = \text{Antilog} (-\text{pK}_a)$$

The percentage error for green indicator (Areca catechu seed extract) obtained from the following relation is tabulated in Table 1:

$$\% \text{ error} = \frac{[\text{OH}^-][\text{H}^+]}{C_A} \times 100$$

[OH]⁻ = Hydroxide ion concentration at end point

[H]⁺ = Hydrogen ion concentration at end point

$$C_A = \frac{C_A^0 V_A}{V_A + V_B},$$

C_A⁰ = Concentration of acid

V_A = Volume of acid

V_B = Volume of base

The colors of green chemicals in the different environments as reported by Patil et al., [25] are shown in Table 6. The color of the green chemicals present in the plant extract species mainly depends on the methoxyl and hydroxyl groups. The color of the plant extract species also depends on the solvent and pH [26].

Areca catechu seed extract possesses natural pigments in their moiety. Areca catechu seed extract mainly contains Catechin, gallic acid, anthocyanin (pelargonidin), epigallocatechin gallate, epicatechin, epigallocatechin, and tannic acid [Fig 6] in their moieties and changes its structure at different pH. As a result of this, the color change of the solution takes place. The factors that contributed to the color variation could be ionic strength, temperature, solvents, and colloidal particles. Another reason may be the chemical composition of the green indicators. Tannins and Flavonoids can show color changes at different pH values as compared to non-tannins and non-

Table 6. Color of the green chemicals in an acidic and basic environment

Phytochemicals	Color with aqueous NaOH	Color with concentrated sulfuric acid	Color with Mg-HCl
Leucoantho cyanins	Yellow	Crimson	Pink
Iso flavones	Yellow	Yellow	Yellow
Flavanones	Orange (Cold)	Crimson	Red, magenta
	Red to purple (hot)	Orange	Yellow
Flavonols	Yellow to orange	Yellow to orange	Red to magenta
Flavones	Yellow	Yellow to orange	Yellow to red
Anthocyanins	Blue-violet	Yellow-orange	Red (fades to pink)

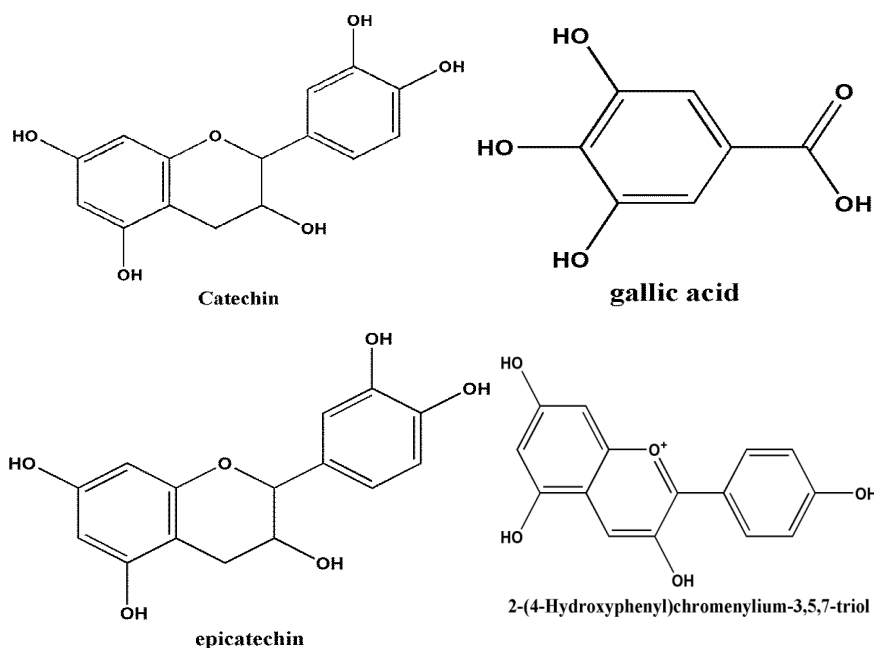


Fig. 7. Main colorants present in Areca catechu seed extract

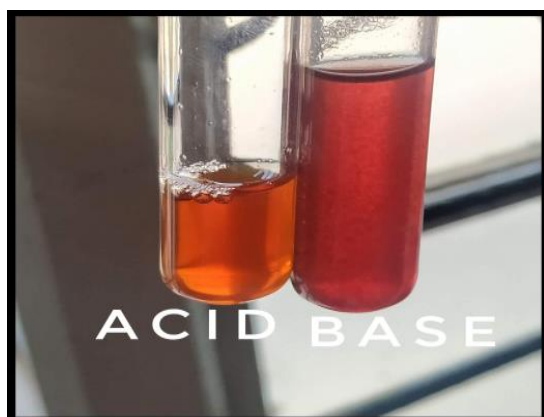


Fig. 8. A color change of Areca catechu seed extract in acidic and basic media

flavonoids derivatives because of the presence of hydroxyl functional groups in tannins and flavonoids.

The colorant present in the Areca catechu seed extract shown in Fig. 7. The change in the color of the solution reflects the structural modifications of the natural pigments which are very sensitive to the pH variation. Depending upon the acidic or basic environment, the natural pigments present in the Areca catechu seed extract exhibit different structure which is responsible for the color change in the solution. The color change (yellowish in acidic and reddish in basic media) is mainly

being due to the presence of electron-rich groups in the Areca catechu seed extract [Fig. 8]. The % error for Areca catechu seed extract is also very less, this strongly supports the usage of Areca catechu seed extract as an effective indicator for various acid-base titrations. Hence, from the current study, Areca catechu seed extract act as a good substitute indicator for synthetic phenolphthalein indicator due to its good performance, accurate results, and simple preparation method.

CONCLUSION

In short, in the current investigation, we exploited a new eco-friendly acid-base indicator with the very good eligibility and category. Areca catechu seed extract exhibits an absorption band at λ_{\max} 280 nm. Results of acid-base titrations show that a toxic phenolphthalein indicator successfully replaced by eco-friendly Areca catechu seed extract. Because, Areca catechu seed extract is easily available, precise, cheap, and accurate. The color change at endpoint due to the presence of pH-sensitive species in the extract and can be prepared before the titration by the Soxhlet extraction process. The best pH range observed in the HCl vs. NH_4OH titration (pH= 3.70 to 8.08). The Areca catechu seed extract performs well for the strong acid-weak base system compared to the other three acid-base titrations. The disadvantage

of an extract of Areca catechu seed is that it must be prepared freshly because Areca catechu seed extract susceptible to the biological side reactions after the 72 hours at 303 K. Hence, it should be stored in the refrigerator to prevent any physical, chemical and biological side reactions before use.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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