Journal Homepage: -www.journalijar.com

INTERNATIONAL JOURNAL OF ADVANCED RESEARCH (IJAR)

Article DOI:10.21474/IJAR01/19450
DOI URL: http://dx.doi.org/10.21474/IJAR01/19450

RESEARCH ARTICLE

SYNTHESIS OF SOAP FROM EDIBLE OILS USING A BIO-BASED CATALYST DERIVED FROM MUSA BALBISISANA COLLA AND A COMPARATIVE STUDY OF QUALITY PARAMETERS

Trisha Dutta, Monmi Saikia and Longim Taro
Department of Chemistry, Eastern Karbi Anglong College, Karbi Anglong, Assam, India.

Manuscript Info

Manuscript History
Received: 10 July 2024
Final Accepted: 14 August 2024
Published: September 2024

Key words:-
Edible Oil, Saponification, Musa Balbisiana Colla, Banana Ash, TFM Value, Quality Parameters

Abstract

Soaps were synthesized by mixing various edible oils such as coconut oil, mustard oil, vegetable oils, olive oil, and soybean oil using banana ash of banana (*Musa balbisiana Colla*.) peel (BPA) as a bio-based catalyst. To improve color, odor, and other qualities; controlled quantities of fillers, permissible natural colors, and fragrances were added. Soaps prepared from edible oils were compared with popular soaps in terms of several parameters such as % yield, TFM (total fatty acid matter) value, total alkali content, free caustic alkali content, pH, and acid value. It was found that the TFM value of edible oil soap is more than 65% for maximum cases; two of them, which have been prepared using coconut oil and olive oil, have TFM value of more than 75%. According to BIS norms, these two types of soap can be categorized as Grade I soap, and the rest are Grade II soaps and it can be used for general bathing purposes. Also, total alkali content, free caustic alkali content, pH value, etc. were found within the prescribed value of BIS.

Introduction:-

The archival use of soap as a cleaning agent for the purpose of washing clothes and personal items attests to the process of saponification contribution to its cleaning and emulsifying properties.[1]Saponification is a process where there is a reaction between fats or glycerides with bases. As per the Indonesian National Standard (SNI) 1994, soap is defined as a sodium compound with fatty acids which are used as a body cleanser, in a solid, foamy form, with or without the addition of other fillers which do not lead to any sort of irritation to the skin.[2] The saponification reaction is also regarded as auto-catalytic as the resulting soap is able to dissolve alkali and also capable of dispersing neutral fat oil into colloidal suspension.[3] Apart from the variable soap applications, research into the chemistry underlying its production and usage serves a deeper understanding of the fundamental molecular structures governing its efficacy. This reaction fabricates the metal salts of fatty acids, or soap molecules, as well as glycerol, which is also popularly known as glycerin. During the process of saponification, a base acts as the nucleophile hydrolyzing the ester linkages in the triglyceride structure. The process causes the cleavage of the ester connection which links the fatty acid chain to the glycerol molecule. This causes the release of free fatty acids, which react with the alkali to produce a soap molecule.[1]The base leads to the ionization of the fatty acid which causes the portion to turn into a salt because of the presence of a basic solution of the NaOH or any other sodium and potassiumcontaining bases. The resulting salt is then precipitated from the solution using a saturated NaCl solution.[4] The two significant constituents of soap are fatty acids and alkali. One of the determining factors for the characteristic of soap is the selection of the type of fatty acids because each type of fatty acid gives rise to different

Corresponding Author:-Monmi Saikia
Address**:-**Department of Chemistry, Eastern Karbi Anglong College, Karbi Anglong, Assam, India.

properties of the soap.[5]The main components of fat and oil composites are the fatty acids, so the choice of the kind of oil which is to be used as a raw material for making soap is very significant.[5]Fats and oils can be classified into three classes, fixed oils, mineral oils, and essential oils. Fixed oils constitute the main raw materials for the fabrication of soap as they cause the decomposition into fatty acids and glycerol when heated strongly, and can be easily saponified by alkali. Fixed oils, include both animal and vegetable fats and oils, which can also be divided according to its physical properties as (a) Nut oils (b) Hard Fats (c) Soft Oils. Nut oils are characterized to have a large proportion of fatty acids with low molecular weight, especially lauric and stearic acid. Examples of these oils include coconut oil. The hard fats contain appreciable quantities of palmitic acid and stearic acids. Examples of these oils include palm oil, animal tallow, and hydrogenated fats. These oils produce slow-lathering soaps but the lather produced is more resistant over long periods of time than the nut oils. Soft oils have substantial amounts of unsaturated acids, namely oleic, linoleic and linoleneic acids. The soap fabricating properties of these oils vary with their fatty acid composition, and their physical and chemical properties of the acids. Examples of these kinds of oils are fish oil, olive oil, cotton seeds and ground nuts. These oils cannot produce a very hard soap when used alone for the fabrication of soap.[4]The article deals with the fabrication of soap using three different oils namely coconut, olive and mustard oil anddifferent catalysts like NaOH, KOH, and catalyst prepared from banana ash**.**

Experimental Section:

Methods of preparation of soap:

10 mL of saturated brine solution was taken in a beaker and cooled in an ice bath. 2 mL of coconut oil and 100 mg of BPA were taken in another beaker and heated at approximately 60-70 °C. Both the mixtures were combined and mixed until set. 2 mL of 95% ethanol was added to the above mixture. Now, the mixture was heated gently for about 10 minutes with constant stirring with a glass rod. The mixture was filtered in a hot condition when bubbles appeared. Then the filtrate was heated again to get a thick gel. The resultant impure soap is goopy and yellow gel. 5 mL of distilled water was added to the mixture and stirred the solution to dissolve the soap and heated the solution to aid in the dissolution process. After getting a clear solution, the soap solution was allowed to cool in the beaker for 5-10 minutes. 5 mL of saturated ice-cooled brine solution was added to the soap solution and stirred well. Pure soap crystals were precipitated out of the solution. The soap crystals were filtered and dried. Tested the soap and the filtrate collected in the flask with pH paper.[6]

Preparation of the catalyst:

The collected *Musa balbisiana* Colla fruit peel was washed with water to remove adhered impurities. The washed parts of the peel were cut into small and thin pieces. Sliced pieces were dried in an oven for 5-6 hours. The dried peel was then burnt in the open air to get ash powder and allowed to cool. The materials were then ground with mortar and pestle, and the powder materials were thereafter stored in an air-tight separate container for further use. This is our catalyst banana peel ash (BPA), which was kept in a desiccator.

Characterization of the Catalyst:

FESEM Analysis

The FESEM images of the catalyst displayed the external morphology of the catalyst (Fig a-f). The images found in varying magnification showed various irregular and non-uniform polygon-like agglomerated structures and many long cylindrical shaped structures. In the catalyst, some distinct structures were seen possessing hexagonal and cuboidal shapes. The image at 100 μm magnification revealed some spongy cluster-like structure whereas the rough texture of sintered ash was also evident from figure (e). At a magnification of 2μm, some light-tiny packed fragmented needle-like strips and clustered particles are also observed.

Determination of alkali content in the soap samples:

5 g of soap sample dissolved in 100 mL hot water. About 40 mL of 0.5N of HNO_3 was added to make it acidic. The mixture was heated until fatty acids floated as a layer above the solution. It was cooled in ice water to solidify the fatty acids. The fatty acids were separated and the aqueous solution was treated with 50 mL of chloroform to remove the remaining fatty acids. The aqueous solution was measured and 10 mL of it was titrated against 0.5N NaOH using methyl orange as an indicator, and from the titre value the alkali content was calculated using the following method.[6]

Calculations:

Total volume of the aqueous solution = V mL

10 mL of aqueous solution required t mL of NaOH

V mL of aqueous solution requires = V× t / 10 = A mL
Amount of NaOH required by acid in aqueous solution = A mL
Volume of HNO_3 required (B mL) = A× Normality of NaOH / Normality of HNO_3
Volume of HNO_3 required for neutralizing NaOH = C = 8 – B
Amount of NaOH in 1000 cc of soap solution (E) =(C × 8 × Normality of HNO_3 g) / 1000
250 cc of soap solution contains (F) = (E × 250) / 1000 g
2 NaOH → Na_2O + H_2O
80 g of NaOH requires (Y) = (62 × F) / 80 g of Na_2O
Weight of soap taken = 1 g
% of alkalinity = (Y × 100) / w = -------------%

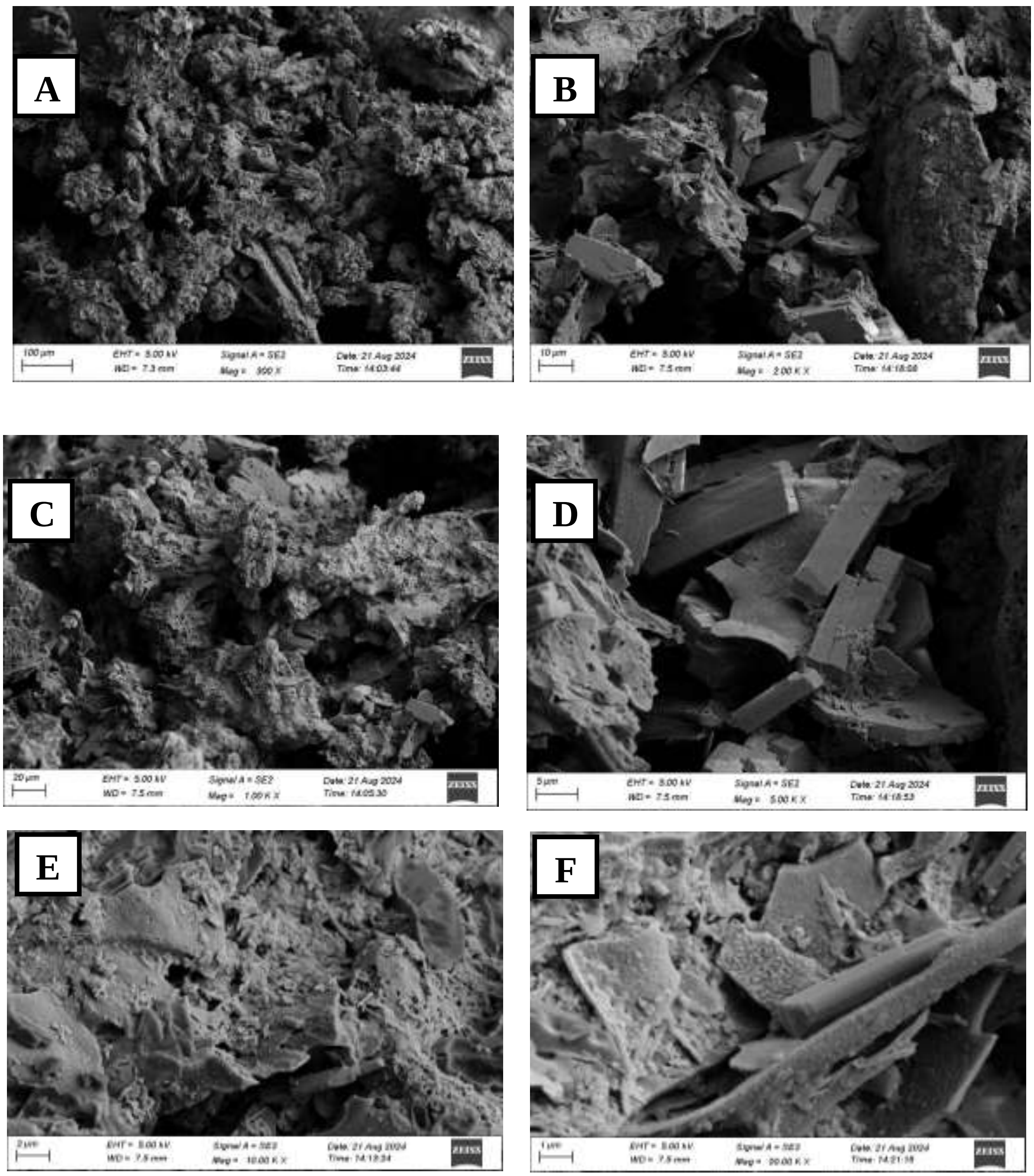

Fig 1:- FESEM images of the catalyst (A-F).

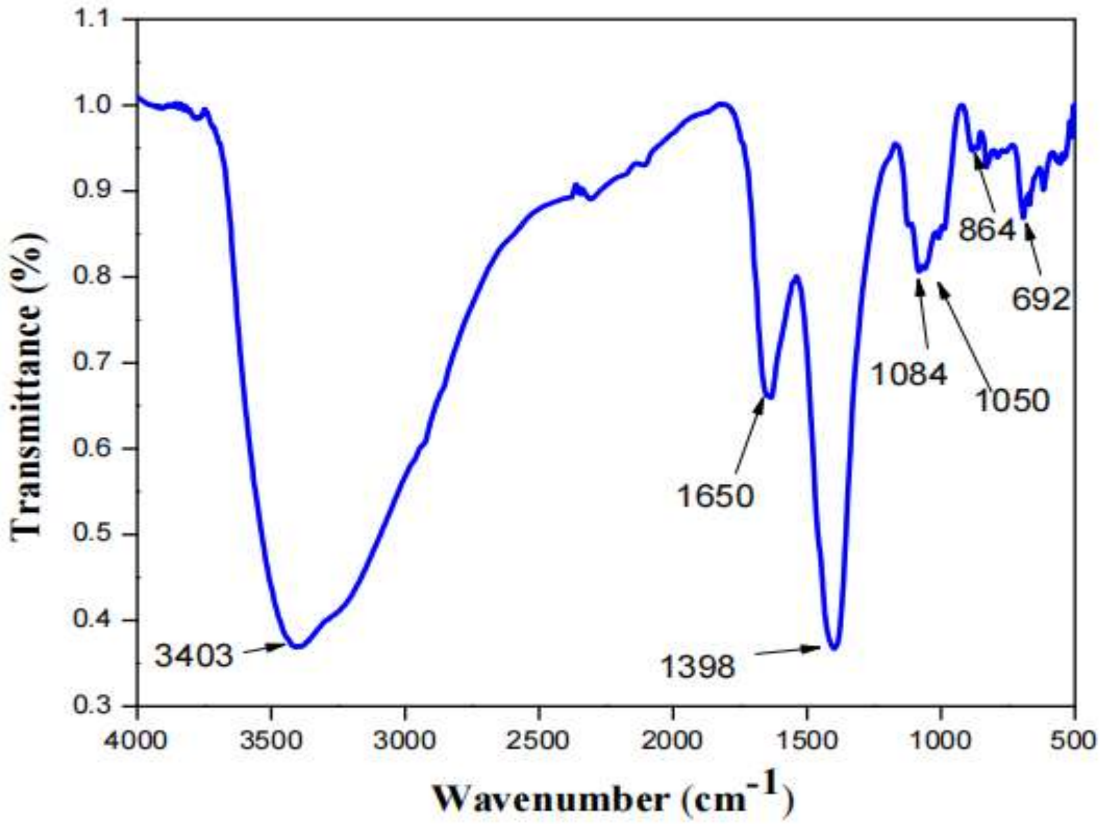

Fig 2:- FT-IR spectrum of the ash catalyst.

The IR image of the ash of the catalyst is shown in figure (2). The peak at 3403 cm^{-1} is attributed to the adsorption of moisture on the surface of the catalysts. The various absorption bands observed in between the ranges of 1650–1050 cm^{-1}is due to the bending and stretching vibrations of C–O bond of carbonate ions of metal carbonates whose formation could be by the adsorption of atmospheric CO_2 on the surface of the metal oxide. These peaks indicated the existence of Ca, K and several other metal carbonates present in the ash catalyst. The peak at 1084 cm^{-1} is due to the presence of Si–O–Si bonds.

Determination of the Total Fatty Matter in the soap:

One of the most crucial elements defining the quality of soap is its total fatty matter (TFM), which is always mentioned in business dealings. When a sample is split with a mineral acid, usually HCl, the total amount of fatty materials, predominantly fatty acids, that can be isolated from the sample is the definition of it. This is the approach and concept that we employed in this instance to ascertain the total fat content of soaps. In terms of total fatty matter, soaps are ranked. TFM is typically linked to soaps that are harder and of lower quality. According to the law, toilet soap is a cosmetic, and it needs to meet all applicable Indian standards. Toilet soaps were divided into three groups by BIS according to the amount of total fatty matter in each. TFM above 76% indicates grade I, or good quality, TFM over 60% is classified as grade II, and TFM over 50% as grade III. International Standards (ISO) state that soaps of superior quality must have a TFM greater t g of soap was taken and transferred into a 50 mL beaker. 20 mL of hot water was added to dissolve the soap. 8 mL of 0.5 N HNO_3 was added to the mixture until the contents were slightly acidic. The mixture was heated over a water bath until the fatty acids floated as a layer above the solution. The mixture was cooled in ice water to solidify the fatty acids and separated. 10 mL of chloroform was added and transferred into a separating funnel. The solution was shaken and allowed to separate into two layers, and the bottom layer was drained out. 10 mL of chloroform is added to the remaining solution in the separating funnel. The fatty acid was dissolved in the chloroform and was again separated as in the previous case and it was transferred to the collected fatty matter. The fatty matter was weighed in a previously weighted petri dish, from the difference in weight the % of fatty matter was calculated in the given soap sample.[6]

Calculation:
Weight of the China dish (x) = _____________
Weight of China dish + Soap after drying (y) =______________
Weight of soap sample = 5 g
% of fatty mater =(y-x)×100Weight of soap sample = _____________

Moisture content:
1 g soap sample was put into a petri dish and placed in an oven for 1 hour at 110^0 C. It is allowed to cool down and down and then weighed. The moisture content in percentage was calculated. The % moisture was calculated using the following formula.[7]
% Moisture = CS-ChCs-Cw ×100,
Cw= weight of crucible, Cs= weight of crucible + sample, Ch= weight of crucible + sample after heating.

Lathering power:
2 mL of water was added to four test tubes and an equal amount of soap solution was added to one test tube of water and shaken vigorously by placing a stopper in the tube. This should give a permanent lather that lasts for at least 30 sec. If the lather doesn't last, then additional soap solution was added to the test tube and shaken vigorously. An equal amount of detergent solution is added to another test tube of water and shaken vigorously. This should give a permanent lather. If not then another 10 drops were added and shaken vigorously. 2 mL of calcium chloride solution to each of the two remaining test tubes of water was added. An equal amount of soap solution to one of the tubes containing calcium ions was added and shaken vigorously. It was observed whether this solution forms a permanent lather and it was noted whether there was any flocculent precipitate in the tube. An equal amount of detergent solution was added to the other tube containing Ca^{2+} ions and shaken vigorously to get a permanent lather.[6]

Cleansing power:
A drop of used brake oil was placed on four separate thin strips of filter paper. It was made sure that the strips of filter paper would fit in the test tubes used in the previous step. One filter paper with an oil spot was placed in the tube containing soap in water. Another was placed in the tube containing detergent and water, a third strip was placed in the tube containing soap in calcium solution, and the fourth strip of oily paper was placed in the tube containing detergent soap, and calcium solution. Each one was shaken well and made sure that the filter paper was immersed in the solution. After 2 min the filter paper was removed and rinsed with tap water. The oil got washed out of the filter paper strip. The solutions were thrown in the sink. The paper strips were thrown in the trash can. The cleansing power of soap versus detergent was compared.[8]

Acid value:
10 mL of ethanol was boiled in a water bath for a few minutes. 2 drops of phenolphthalein and 0.1 N NaOH are added to produce a permanent pale pink colour. Then 1 g of oil was added to 10 mL of this neutralized solution and the mixture was boiled on a water bath. While still hot the solution was titrated against 0.25N NaOH until the pink colour returned.

Determination of pH:
pH of aqueous edible oil soap solution (5%) was recorded by using pH paper as well as pH meter. Similarly, pH of various branded soaps was recorded.[8]

Results and Discussion:-
A total of 10 numbers of soap were synthesized using 10 different edible oils under standard reaction conditions. The yield obtained was found to be the maximum for olive oil, as shown in the table 1.

Fig 3:- Soap obtained from mustard oil

Fig.4:- A few soaps from different edible oil.

Few more samples of soap were prepared by following the same procedure along with addition of natural color extracted from Neem leaves, Rose flowers and Hibiscus flowers. An image of a soap made of olive oil and neem leaf extract is shown below:

Fig.5:- Soap of Olive oil and Neem leaf extract.

The addition of natural color, which has medicinal value, increases the quality of the soap.

Table 1:- % yield of soap using individual oil:

Serial No	Soap Name	Oil	Catalyst	Weight of soap (in g)	% of yield
1	Soap 1	Coconut oil	BPA	1.4	90
2	Soap 2	Almond oil	BPA	1.68	84
3	Soap 3	Soybean oil	BPA	1.6	80
4	Soap 4	Refined oil	BPA	1.152	84.8
5	Soap 5	Rice bran oil	BPA	1.696	79.23
6	Soap 6	Til oil	BPA	1.5154	75.77
7	Soap 7	Sunflower oil	BPA	1.5658	78.29
8	Soap 8	Mustard oil	BPA	1.34	66.96
9	Soap 9	Olive oil	BPA	1.882	94.1
10	Soap 10	Vegetable oil	BPA	1.728	80.4

A comparative study using the standard protocolhas been performed taking olive oil, where the catalytic activity of BPA was compared with a few conventional base catalysts (table 2). The reaction time was kept the same for all the catalysts. It was found that soap using BPA gave a comparatively high yield.

Table 2:- Comparison of the BPA catalyst with other conventional base Catalysts.

Serial No	Oil	Base	Weight of soap (in g)	% of yield
1	Olive oil	NaOH	1.4	90
2	Olive oil	Na_2CO_3	1.39	69.5
3	Olive oil	KOH	1.666	83.3
4	Olive oil	BPA	1.882	94.1
5	Olive oil	K_2CO_3	1.6	80

TFM values were calculated according to standard formula.[8]According to Bureau of Indian Standards (BIS) norms, soaps having TFM value minimum 75% are considered as grade I soap, they are the safest for the skin. They are also the most superior in terms of cleansing and moisturizing properties. Soaps with a TFM content between 70% and 75.99% are Grade II and are considered good quality but slightly less superior than Grade 1 soaps. The soaps with a minimum TFM of 60% are considered Grade III soap. Here, we found that soaps prepared from olive oil and coconut oil can be considered as grade I soap having a high TFM value. Again, the TFM values of the soaps prepared in this study were compared with commercially available soaps like cinthol, dettol, lifebuoy and lux; where soaps prepared from olive oil and coconut oil showed comparatively better quality than the others (table 3).

Table 3:- Total Fatty Matter Content on Soap Samples.

Sl No	Name of soaps	Weight of China Dish (g)	Weight of China Dish+ soap after drying (g)	% of fatty matter
1	Soap 1	20.2	25.07	77.4
2	Soap 2	20.2	23.89	73.8
3	Soap 3	20.2	23.675	69.5
4	Soap 4	20.2	23.08	57.6
5	Soap 5	20.2	23.33	62.6
6	Soap 6	20.2	22.975	55.5
7	Soap 7	20.2	23.075	57.5
8	Soap 8	20.2	23.175	59.5
9	Soap 9	20.2	24.15	79
10	Soap 10	20.2	23.7	70
11	Lifebuoy[8]	-	-	63.4
12	Lux[8]	-	-	72.0
13	Cinthol[8]	-	-	77.8
14	Dettol[8]	-	-	72.2

According to the Bureau of Indian Standards (BIS), good quality soaps should have less than 5% alkali content. The ISO specification, however, states that soaps should have less than 2% alkali content. [6,7]

Table no 4:- Total alkali contents in the soap samples.

Sl No.	Name of Soap	Volume of Soap (ml)	Burette Reading(ml)NaOH		Volume of NaOH (ml)	Total Vol of Aqueous Solution	% of alkalinity
			Initial	Final			
1	Soap 1	10	0	1	1	138	2
2	Soap 2	10	0	0.4	0.4	140	3
3	Soap 3	10	0	0.5	0.5	138	2.6
4	Soap 4	10	0	0.3	0.3	135	2.76
5	Soap 5	10	0	0.7	0.7	132	2.48
6	Soap 6	10	0	0.6	0.6	140	3.16
7	Soap 7	10	0	0.3	0.3	138	2.8
8	Soap 8	10	0	0.7	0.7	138	2.4
9	Soap 9	10	0	1.3	1.3	140	1.6
10	Soap 10	10	0	0.4	0.4	128	2.7

The alkali mainly used is a soluble salt of an alkali metal like sodium or potassium. The alkalis used in soap making are NaOH (sodium hydroxide) and KOH (potassium hydroxide). Sodium carboxylates are the common toilet soap. Potassium carboxylates or potassium soaps are obtained when saponification of a fat or oil is carried with potassium hydroxide.[6] Potassium soaps are softer than sodium soaps and they are used for special purposes when rapid solution is desired eg: in making shaving creams or liquid soaps. It was reported that BPA (peel of *Musa balbisiana*

*Colla*Ash) has highest quantity of potassium carbonate as compared to sodium carbonate, which is cheap and renewable sources of potassium carbonate.[9] Soaps prepared using BPA as catalyst have marginal alkalinity values as well as a standard TFM value as shown in Table 4 and Table 3.

It was also found that soaps prepared from coconut oil and olive oil have good lathering and cleansing power (table 5).

Table 5:- Lathering and cleansing power.

Serial No	Lathering power	Cleansing power
Soap 1	Very good	High
Soap 2	Good	Moderate
Soap 3	Good	Moderate
Soap 4	Good	Moderate
Soap 5	Poor	Poor
Soap 6	Poor	Poor
Soap 7	Poor	Poor
Soap 8	Moderate	High
Soap 9	Moderate	Moderate
Soap 10	Very good	High

The pH of the soaps prepared are taken using pH paper and pH meter, in both cases we found the approximately same value for each type of soap. It was observed that the soaps prepared from coconut oil and olive oil have mild basic character, which are comparable with some commercially available soaps (table 6).

Table 6:- pH value of soaps.

Sl No	Name of soaps	pH range as (indicated by pH paper)	pH range as (indicated by pH meter)
1	Soap 1	7-8	7.8
2	Soap 2	8-9	8.65
3	Soap 3	8-9	8.5
4	Soap 4	8-9	8.6
5	Soap 5	8-9	8.3
6	Soap 6	8-9	8.7
7	Soap 7	8-9	8.6
8	Soap 8	8-9	8.1
9	Soap 9	7-8	7.4
10	Soap 10	8-9	8.6

Moisture content is a measure of the amount of free water present in the soap. It is an important parameter used to determine the shelf life of soap. Excess water in soap reacts with any unreacted triglycerides (Fats/Oils) which might be present in soap, that is, they undergo hydrolysis to form fatty acids and glycerol on storage. Overall, the percent of moisture content values determined in all the soaps were within acceptable limits (table 7). The lower acid value of the prepared soap indicates a good cleansing power.

Table 7:- Moisture content and acid values.

Serial no	Moisture content (%)	Acid value
Soap 1	14.6	1.4
Soap 2	17.17	1.6
Soap 3	19.74	2.1
Soap 4	16.64	2.4
Soap 5	18.54	2.7
Soap 6	17.87	2.4
Soap 7	14.76	2.7
Soap 8	21.04	2.6
Soap 9	14.40	1.3
Soap 10	19.43	2.4

Soaps are graded in terms of total fatty matter or TFM. TFM is a measure for identifying the amount of fatty matter present in soaps. The TFM measures the quality of soap and the accepted percentage value for toilet soap is between 76-77%. The best blend is selected on the basis of TFM.

Conclusion:-

In summary, this study ascertained that the soaps prepared from olive oil and coconut oil have low alkali content and high fatty matter, they are considered as good quality soap. The overall fatty matter is linked to soap quality, percentage of which predicts the soap as harder and softer and it is the most crucial feature characterizing the soap's quality. The other soap samples that we prepared from different vegetable oils are all within the typical TFM value range, out of which soaps prepared using coconut oil and olive oil have the best quality. The use of a bio-based catalyst BPA enhances the quality of soap and makes the soap-making procedure environmentally benign. By studying all the parameters like total fatty matter, alkali content, moisture content, lathering power, cleansing power, and acid values of all prepared soap; it was found that the soap prepared from coconut oil and olive oil using BPA has good detergent quality. All these data indicate good detergent quality, which lies in the range of toilet soaps. These soaps have excellent lathering power and cleansing power.

Acknowledgement:-

Authors thank the support of SAIC, IASST, Guwahati for FESEM analysis.

Conflict of Interest:
There is no conflict of interest regarding this article.

Supplementary Information:
All the spectroscopic data are enlisted in the supplementary section.

References:-

1. Alum, B.N.Saponification Process and Soap Chemistry.
2. Sukeksi, L.; Diana, V.J. Phys. Conf. Ser. **2020**, 1542(1),012046.
3. Saxton, K.; Crosby, B.; Dunn, K.J. Sci. **2013**, 2.
4. Abeyah, O.J.; Aina,E.A.; Okuonghae. JournalofPureandappliedsciences. **1998**,17-23
5. Widyasanti, A.; Ginting, A.M.L.; Asyofami, E.; Nurjanah, S.IOP Conf. Ser.: Earth Environ. Sci.**2018**, 141(1), 012037
6. Betsy, K.J.; Jilu, M.; Fathima, R.; Varkey, J.T.Asian J. Appl. Sci. Technol.**2013**, 2 (1), 2249-0698, pp. 8-12
7. Mak-Mensah, E.E.; Firempong, C.K.Asian J Plant Sci Res, **2011**, 1 (4):1-7
8. Sarasan, G.; Rangwala, J.A. Int. J. Chem. Sci.**2014**, 12(1), 306-314
9. Deka, D.C.; Talukdar, N.N.I.J.T.K.**2007**, 6(1), 72-78.