SEMI ORGANIC DETERGENT PRODUCTION



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DEPARTMENT OF CHEMICAL ENGINEERING ASSAM ENGINEERING COLLEGE GUWAHATI - 781013 AUGUST 2024 – NOVEMBER 2024

SEMI ORGANIC DETERGENT PRODUCTION

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Date:

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ABSTRACT

Detergents are rapidly growing products in FMGC due to urbanisation and upgrades in the lifestyle of the major population. In India, approximately 6 million metric tons of detergents is consumed annually. Simultaneously, the environmental impacts such as water and soil pollution due to the usage of detergents has also been increasing rapidly. The aim of this project is to replace some of the raw materials generally used in detergent production with other organic substitutes.

The first substitute is sodium silicate produced from rice husk ash (RHA). The RHA was obtained by burning rice husk in muffle furnace and then its composition analysis was carried out using FTIR spectroscopy. The RHA was then reacted with sodium hydroxide (NaOH) solution of 1M and 2M concentration to get sodium silicate. Solubility test and the pH test were then performed for the produced sodium silicate.

The second organic compound that was incorporated into the production process was ashes of banana stem. Banana stem was first dried and then burnt to get the ash. The ash was then mixed with water in a beaker and then heated over a hot plate to remove the impurities along with the water. At last white powders – soda ash (Na_2CO_3) remained in the bottom of the beaker.

The last substitute was the use of coconut oil which provided the fatty acids required for the detergent production process. Furthermore, the detergent samples produced were analysed for quality parameters such as the pH value, foam stability, moisture content and the free alkalinity test and the compared with those of an industrially produced detergent.

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CHAPTER 1 INTRODUCTION

1.1. Detergent

Detergent is a water-soluble cleaning agent that combines both impurities and dirt to make them soluble, unlike soap; its variant is not forming a scum with salts in hard water. Often, we use the words soap and detergents interchangeably, but they are two different things. Barlass mentioned that detergents are chemical substances that break up and remove grime and grease in the same matter as soap but remain efficient even in hard water. In hard water, soap form insoluble compounds with the magnesium and calcium ions which precipitate out and hinders the foaming and cleaning potential of soap. In contrast, detergents react with the ions in hard water to form soluble compounds or remain colloidal to be dispersed. Nowadays, detergents are synthetic chemicals mixture and additives formulated in a huge chemical plant. The use of detergent is span across from shaving foam to hair shampoo, clothes washing powder to stain removal, etc. David presents the molecular structure of soap as presented below:

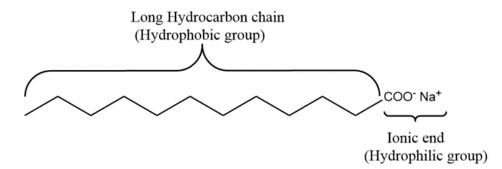


Fig. 1.1: Basic soap structure

On the other hand, detergents are structurally similar to soaps, but the disparity is in the water-soluble section. The two examples of detergents are shown as:

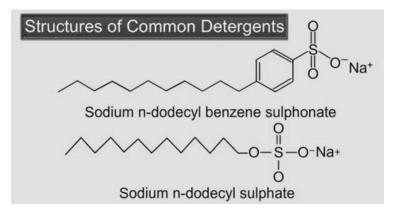


Fig. 1.2: Basic detergent structure

Detergents are amphipathic molecules that have hydrophobic and polar groups. In these molecules, the polar group is at the end of the hydrophobic carbon chain. Unlike the purely polar or non-polar molecules, the amphipathic molecules show unique chemical properties in water. In water, the polar group in detergent forms hydrogen bonds with water molecules, while the hydrophobic reactions aggregate the hydrocarbon chains. These abilities allow detergents to be soluble in water. Besides, when detergent or soap is in water, their molecules clusters and it referred to as micelles. Then, the polar ends of the molecules are on the outside of the cluster, as the non-polar ends are in the middle. In aqueous forms, they form spherical structures known as micelles in which each contains numerous detergent molecules. According to Bhairi and Mohan, detergents can solubilize hydrophobic compounds in water due to their amphipathic nature. Again, detergent is also known as surfactant, as a surfactant reduces the surface tension of water. Most times, the substances are alkylbenzene sulfonates – a group of compounds that are like soap but can dissolve in hard water. In these compounds, the detergent's polar sulfonate is less probable than the soap's polar carboxyl to bind with calcium and other ions in hard water.

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1.2. Industrial Detergent production and Problems associated

The main raw material used for industrial detergent production is Linear Alkyl Benzene Sulphonic Acid (LABSA). They act as a surfactant and are the main component of the detergent production process. The other main components are fatty alcohols and caustic soda which when added to LABSA produce the sodium salts. The resultant sodium salts are mixed with detergent builders to give the final detergent product.

Detergent builders are the auxiliary materials that are added to detergents to enhance the activity and cleaning capability of a detergent or surfactant material. These generally include sodium tripolyphosphate (STPP) – used to soften hard water and produce foam; and sodium silicates – used as corrosion inhibitor and to increase cleaning power. Other builders include soda ash (for water softening and pH adjustment), hydrogen peroxide (for disinfecting and whitening clothes), ammonia (for emulsifying grease and oils) and several such compounds.

The main problem associated with detergent production is the use of synthetic raw materials. The use of such materials has led to non – biodegradability of the detergents after use. The foam produced remain on the surface of the water as it passes through the sewage systems into the rivers. With the growing use of detergent, the chances of water pollution are also increasing which should be tackled as soon as possible.

1.3. Project Objective

- Promote Waste utilisation: Transform rice husk, an abundant agricultural byproduct and banana stem, into a valuable resource, minimizing environmental waste and contributing to sustainable waste management, thereby reducing reliance on synthetic chemicals.
- Develop Eco-Friendly Detergents: Formulate a detergent with a reduced environmental impact by integrating organic components derived from rice husk, banana stem and coconut oil with conventional detergent ingredients.
- Enhance Cleaning Efficiency: Ensure the resulting semi-organic detergent meets or exceeds the cleaning performance of traditional detergents while remaining safe for both users and the environment.

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This project seeks to bridge the gap between effective cleaning solutions and environmental sustainability, demonstrating the potential of rice husk and banana stem as an innovative and eco-friendly alternative in detergent production.

1.4. Project Scope

The project will design and test scalable production methods to ensure quality and consistency while assessing the cleaning efficacy of the detergent on various stains and fabrics. Comparative studies will evaluate its performance against conventional and organic alternatives. A critical part of the project is analysing the environmental impact by examining the detergent's biodegradability and its potential to reduce agricultural waste. Additionally, the project will explore the economic feasibility, including production costs and market potential, to ensure the product is competitive and accessible to consumers.

Sustainability will be a central theme, with efforts to promote the use of agricultural byproducts and reduce reliance on non-renewable resources. By transforming rice husk and banana stem into a value-added product, this project aims to address waste management challenges while contributing to the development of sustainable and effective cleaning solutions.

CHAPTER 2 LITERATURE SURVEY

The reviewed studies focus on utilizing rice husk ash (RHA), a byproduct of rice milling, to produce sodium silicate, which is then used in detergent manufacturing. This approach addresses environmental challenges related to RHA disposal while providing a cost-effective alternative to industrial sodium silicate. All studies employ chemical reactions involving sodium hydroxide (NaOH) and RHA, analysing the resulting sodium silicate's performance in detergent production. Numerous technical papers on the synthesis of detergents using organic components have been presented at the research level. We referred to many of these papers for our study, which are listed below:

- Parth Shah and Setu Visvadiya's study [Reference 2] highlights that mesoporous silica, a crucial raw material for various applications, can be derived from paddy husk ash. In detergent production, silica plays a significant role in the synthesis of sodium silicate. His findings indicate that sodium silicate can be produced through a semi-organic method.
- 2. Perpretua G. Bassey and Emmanuel A. Etim's research [Reference 1] reveals that rice husk, often considered a waste material, contains a high percentage of silica. His study demonstrates that sodium silicate can be effectively produced using silica extracted from rice husk. This silica can then serve as a raw material in combination with other chemicals for detergent production, making the process both practical and efficient.
- 3. Legesse Adane's study [Reference 3] explored the use of palm kernel oil, treated with an alkaline solution, for detergent production. However, he concluded that this method is prohibitively expensive due to the need to import palm kernel oil. Additionally, his research showed that used cooking oils could serve as an alternative for detergent production when treated with sodium hydroxide, offering a more cost-effective solution.

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CHAPTER 3 MATERIALS AND METHODOLOGY

3.1. Organic Materials Utilised

3.1.1. Rice husk

Rice is a staple food for approximately half of the global population and India is one of the leading producers. Rice husk, a byproduct of the milling process, is one of the most widely available agricultural wastes in the country. This husk contains about 75% organic volatile matter and the balance 25% of the weight can be converted into ash, known as rice husk ash (RHA), by firing process. The RHA is predominantly composed of 80 - 95% silica, which is highly porous, lightweight and has a very high external surface area, making it valuable for industrial use.

Due to this unique composition, RHA when reacted with NaOH at around 100°C produces sodium silicate crystals. In this project, the sodium silicate produced form RHA have been used as a builder in the detergent production process. This sodium silicate can be an organic alternative to the industrially produced sodium silicate used in detergent manufacturing.

3.1.2. Banana Stem

Banana plant pseudo stem have been in use as a cleansing agent since time immemorial. The pseudo stem is first dried and then converted into ash by firing processes. Locally this ash is known as "Khar" or "Char" or "Kalchi". This ash has about 30 - 40% soluble component in water which in turn consists of around 80 - 90% of soda ash. In this project we aim to replace the industrially produced soda ash, used a detergent builder, by the soda ash produced from the banana plant stem.

3.1.3. Coconut Oil

Fatty alcohols (derived from fatty acids) are important raw materials for production of detergents. Usually in detergent industries, synthetic fatty alcohols are used which are produced from ethylene using Alfol process. In this project the source of the fatty acid will be coconut oil. Coconut oil is rich in saturated fats, particularly lauric acid, which

is an effective cleaning agent. The fatty acids in coconut oil contribute to its ability to break down grease and oils, making it a powerful detergent ingredient.

3.2. Rice Husk Acquisition and sample preparation

The rice husk used in this project was obtained Nalbari district of Assam. This material was sundried to reduce any moisture content and then sieved to remove any remaining rice grains that may be present in the husk. Then, the rice husk were grinded and then placed into crucibles. The crucibles were then put into furnace at a temperature of 700°C for 6 hours. After this process we obtain rice husk ash (RHA) which is the main component for the production of sodium silicate. FTIR tests were then carried out for both the rice husk and rice husk ash.



Fig.3.1: Grinded Rice Husk

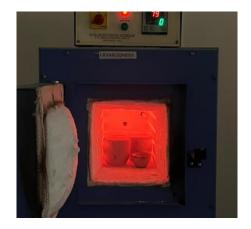


Fig.3.2: Grinded Rice Husk being heated inside the furnace



Fig.3.3: Rice Husk Ash (RHA)

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Thereafter, we prepare two sodium hydroxide (NaOH) solutions, with concentrations of 1M and 2M, were prepared. This was done by adding 40g of NaOH pellets into 1L of distilled water and 80g of NaOH pellets into 2L of distilled water for the 1M and 2M solution respectively and then letting them ferment for 48 hours.

3.3. Production of sodium silicate

To produce sodium silicate 20g of RHA was added to 100mL of each of the NaOH solutions in two separate beakers. The solutions were mixed properly with a glass rod and then put into the hot air oven at a temperature of 100°C for 1 hour. After this, the solution was cooled down and poured into a filter paper. The residues on the filter paper were then dried at a temperature of 60°C for about 30 minutes. The viscous products that we get from this process are the sodium silicates obtained from 1M and 2M NaOH solutions. Afterwards the sodium silicates were tested for solubility and the pH value.

The chemical reaction between RHA's Silicate (SiO₂) and sodium Hydroxide (NaOH) result in formation of Sodium Silicate (Na₂SiO₃) and water (H₂O) represented by:

 $2NaOH + SiO_2 \longrightarrow Na_2SiO_3 + H_2O$



Fig.3.4: *Filtration of* reacted NaOH + RHA



Fig.3.5: Filtered and dried Na₂SiO₃

3.4. Banana stem acquisition and production of soda ash

The banana stems were also obtained from Nalbari district. They were first sundried till the colour of the stems were changed from green to brown, which indicated the removal of any remaining moisture. Thereafter, the dried stems were burnt directly with the help of fire. To know the composition of the obtained ash, FTIR test was carried out for it. The ash was then grinded to fine powders and then mixed in water with the help of a glass rod. The solution was then filtered with the help of a filter paper to remove the impurities (residues). The filtrate was taken in a beaker and placed over a heating plate at a temperature of 80°C to evaporate the water. When the liquid was completely vapourised, a white powdery substance – soda ash was obtained on the bottom of the beaker.



Fig.3.6: Mixing of banana stem ash and water



Fig.3.7: *Filtration of the alkaline solution*

3.5. Detergent Synthesis

The basic components used in the production of the detergent are presented in Table 3.1. 200mL of the fermented NaOH solution (1M) were poured into a bucket. Then 10mL of the sodium silicate prepared from 1M NaOH solution and 500mL of coconut oil were added into the bucket and stirred continuously. 500g soda ash prepared from the banana stem ash and 50g of sodium tripolyphosphate were also added while stirring was going on. Thereafter, 50mL of ammonia and 500mL of hydrogen peroxide were added to the solution and the stirring speed was increased. After this step, an excessive amount of foam was forming which was stabilised by the increased stirring speed. At last, 100g of sulphonic acid was added. The stirring was continued till the foam stabilised and there was no more foam formation. The solution was then left out to dry till the solution. The detergent was strained out of the remaining liquid using a muslin

cloth and then again sundried to achieve the required consistency. Then a sieve was used to reduce the detergent particle size to fine powdery form.

The steps can be summarized as:

Caustic soda \rightarrow Coconut Oil (organic) \rightarrow Soda ash (organic) \rightarrow Dodecyl Benzene Sulphonic Acid \rightarrow Sodium silicate (organic) \rightarrow Sodium Tripolyphosphate \rightarrow Ammonia \rightarrow Hydrogen Peroxide = **DETERGENT**

The same steps were repeated to produce detergent from sodium silicates from the 2M NaOH solution. The amount of product synthesised in grams was found to be 1620g. Afterwards, the properties, namely, foaming stability, stain removal ability, pH value, free alkalinity and moisture content were evaluated and compared with that of the industrially produced detergent.



Fig.3.8: *Mixing of the detergent components*



Fig.3.9: Drying of the mixed solution



Fig.3.10: Straining out the remaining liquid



Fig.3.11: Final drying step



Fig.3.12: *Final detergent product*

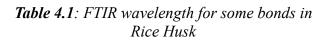
Component	Function	Quantity
Caustic Soda Solution	control of pH levels and	200mL
	increase solubility of detergent	(9%)
Coconut Oil	acts as the fatty acid in	500mL
	detergent production and has	(26%)
	antimicrobial properties	
Soda Ash	pH control, helps in grease	500g
	removal and softens water	(26%)
Ammonia	neutralizes the acid and helps	50mL
	in stain removal	(3%)
Hydrogen Peroxide	Acts as deodorant,	500mL
	brightener/whitener and	(26%)
	disinfectant	
Sodium Tripolyphosphate (STPP)	Acts as water softener, aids in	100g
	foaming and wettability of the	(5%)
	detergent	
Sodium Silicate	Controls viscosity of detergent	10mL
	and acts as corrosion inhibitor	(1%)
Dodecyl Benzene Sulphonic Acid	acts as the surfactant and helps	100g
	in dirt and grease removal	(5%)

CHAPTER 4 RESULTS AND DISCUSSION

4.1. Composition of Rice Husk and Rice Husk Ash

Fig.3.1. shows the rice husk acquired. The composition of the rice husk obtained by FTIR analysis method is mentioned below:

Compounds or bonds	Corresponding FTIR wavelength
C – N	1034 cm ⁻¹
Si – C	790 cm ⁻¹



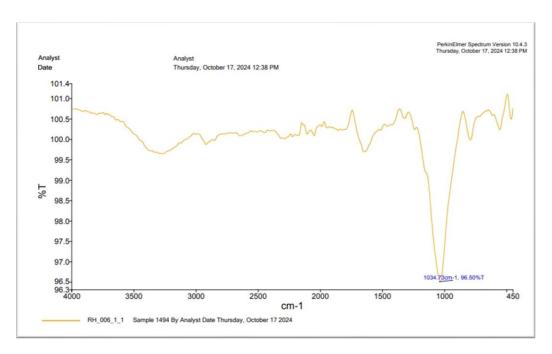


Fig.4.1: FTIR chart for Rice Husk

The composition of the Rice Husk Ash, obtained by the method discussed in topic 3.2, is mentioned below:

Compounds or bonds	Corresponding FTIR wavelength
Si – O	466 cm ⁻¹
Si – C	790 cm ⁻¹

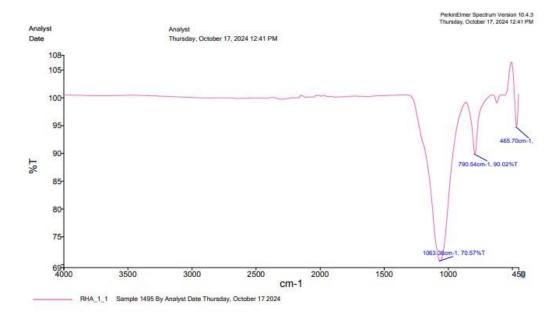


 Table 4.2: FTIR wavelength for some bonds in RHA

Fig.4.2: FTIR chart for RHA

4.2. Formulated Sodium Silicate and its properties

The most abundant element in RHA is Silicate (SiO₂). The solubility test of the formulated sodium silicate (Na₂SiO₃) from RHA in water and 95% w/w alcohol showed that sodium silicate (Na₂SiO₃) was incompletely soluble in both medium, it gave cloudy colour with the homogenous mixture. The cloudy colour was attributed to the presence of other elements in the RHA. Figure 4.3 shows the solubility results from the RHA Sodium silicates (Na₂SiO₃), that is, samples A and B (1M Na₂SiO₃ and 2M Na₂SiO₃ respectively).

The pH tests showed that the pH value from 1M NaOH RHA Sodium silicate (Na₂SiO₃) was close to 11.11, while 2M NaOH RHA Sodium silicate (Na₂SiO₃) was close to 11.66.



Fig.4.3: Solubility test of sodium silicate

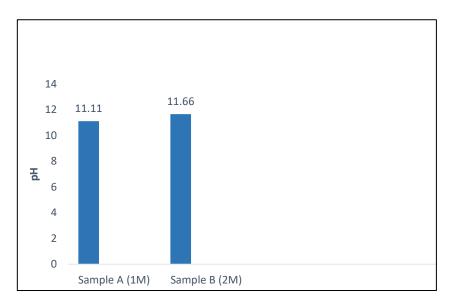


Fig.4.4: pH of produced sodium silicates

4.3. Soda Ash extracted from Banana Stem

Banana pseudo stem ash is typically composed of alkaline minerals such as calcium carbonate (CaCO₃), potassium carbonate (K₂CO₃), sodium carbonate (Na₂CO₃), and other impurities.

The composition of banana pseudo stem ash obtained by FTIR analysis method is mentioned as follows:

Compounds or bonds Corresponding FTIR waveler	
C - H, C - Cl	783.31 cm ⁻¹
С – Н	684.37 cm ⁻¹
C – O	991.28 cm ⁻¹
C = O (carbonates)	1637 cm ⁻¹
C – N	1377.63 cm ⁻¹

Table 4.3: FTIR wavelength for some bonds inBanana Stem Ash

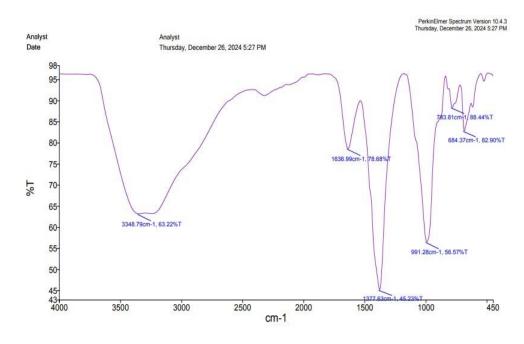


Fig.4.5: FTIR chart for Banana Stem Ash

4.4. Detergent Characterization

The produced detergent samples were characterized by the following tests and the results were compared with that of an industrially produced (Surf Excel) detergent.

4.4.1. Determination of Foam Stability

The detergent produced was used to form lather in water and the time for the foam to collapse was determined using a stopwatch. Generally, the quality of foam produced is what makes soap or detergent different from other products. The foam produced reduces surface tension and makes it possible for cleansing to take place. Thus, foaming

increases when the components of the detergent bound together effectively. Hence, foam stability is the duration with which foaming is sustained when detergent dissolves in water. Figure 4.6 represents the foam stability of the produced detergents from the two different Sodium silicate (Na₂SiO₃) samples:

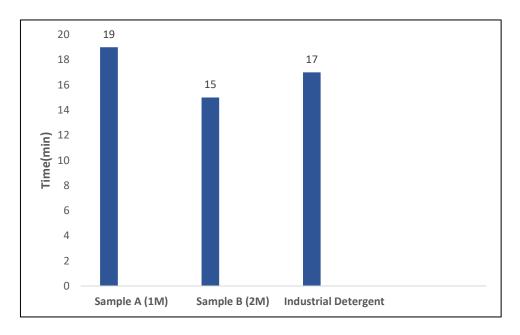


Fig.4.6: Foam Stability of the samples

4.4.2. Moisture Content determination

This is the amount of water present in the detergent sample. Moisture affects the cleansing and lathering property of the detergent. Besides, if moisture is too low, the detergent is liable to take a long time before dissolving in water. If on the contrarily, the detergent components will dissolve and lose its effectiveness in terms of foam formation and may stick to its container.

About 15g of the samples (Sample A, Sample B and industrial detergent) were taken and heated so that the moisture gets removed. After removal of moisture the moisture free detergent samples were weighed.



Fig.4.7: Drying of the samples to calculate moisture

Figure 4.8 shows the weight of the sample after moisture removal. The moisture content was obtained by the following formula:

$$M = \frac{C_w - C_d}{C_w} \times 100$$

where, $M = moisture \ content \ of \ the \ sample \ (\%)$ $C_w = weight \ of \ wet \ sample$ $C_d = weight \ of \ dry \ sample$

The results obtained indicated that the produced detergent from the samples had about the same moisture content. Sample A (1M) had about 10.66% moisture, Sample B (2M) had about 11.93% of moisture content and industrial detergent had about 11.51% moisture content.

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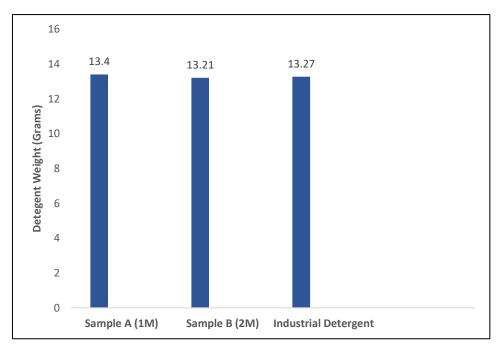


Fig.4.8: Moisture Content of the samples

4.4.3. Determination of pH value

pH is an important quality parameter for detergent. It is the quickest indicator that reveals the progress of a successful detergent making. It determines the friendliness or otherwise of a detergent to the skin. The quality set for detergent in terms of pH based on literature is in the range of 9 - 11. The closer the detergent is to neutrality the better the detergent quality. Thus, 1% of the detergent solution was prepared by dissolving about 0.5g of the produced detergent in 50mL of water. The heat was applied to dissolve the detergent completely. Using a pH meter the alkalinity of the solution was determined by dipping the electrode of the pH meter into the detergent solution and value obtained was recorded.

Figure 4.9 shows that Sample A and Sample B had pH values of 10.38 and 10.45 respectively whereas the industrial detergent has a pH value of 10.61.



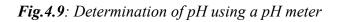
Sample A







Industrial Detergent



4.4.4. Determination of Free Alkali

Free alkali gives an insight into the amount of excess alkali present in the detergent. Free alkali is injurious to the skin; hence the quality of detergent is determined by how free the soap or detergent is from alkali. A sample of the produced detergent (10g) was placed in a conical flask and 100mLof neutralized alcohol (ethanol) was added. The flask and the content were placed on a water bath and heated until the detergent dissolved in the alcohol.

Then, 10cm^3 of Barium Chloride solution and 2 drops of phenolphthalein indicator were added to the conical flask. The whole content was titrated against $0.1 \text{M H}_2\text{SO}_4$ until the solution became colourless. The free alkali was calculated by:

$$A_{free} = \frac{3.1 \times M \times V}{W} \times 100$$

where, $A_{free} = free \ alkali \ in \ \%$

 $M = molarity of H_2SO_4$ solution in mol/L

V = volume of H_2SO_4 solution used in titration

W = weight of the detergent sample in gram



Table 4.10: Titration for the determination of free alkali

It is noted that the produced detergent from the various samples had close free alkali values. This is because the sulphonic acid used in the detergent was not sufficient or enough for the detergent to neutralize efficiently. It was observed from the result that the presence of free alkali increased as a result of the class of components used for the detergent production which could be termed as acidic. Based on the standard specifications, the allowable amount of free alkali content of detergent is of the range 0.00-0.06%. Thus, Sample B (i.e., 2M NaOH RHA Na₂SiO₃) free alkali value was closer to the standard alkali content in the literature than the other sample. This

closeness of the produced detergents" free alkali is further attributed to their pH values obtained, which were within the known range of pH of 9 - 11 for quality detergent.

	Sample A (1M)	Sample B (2M)	Industial Detergent
Foam Stability	19 mins	15 mins	17 mins
Moisture Content	13.4 %	13.21 %	13.27%
pH Values	10.38	10.45	10.61
Free Alkalinity	0.08029 %	0.0651 %	0.07471 %

 Table 4.4: Test results of the produced detergents compared to industrial detergent

In summary, industrial characteristics that are considered to be of utmost importance in the detergent production are free alkali content, pH value, foam stability and moisture content. Among these characteristics, free alkali content and pH value were observed to be affected by the quality of the basic components and the quantity of organic oil used. So far, the results obtained in respect of all these parameters indicated that the detergent was within acceptable specification and quality.

CHAPTER 5 CONCLUSION

The production of detergents using rice husk, banana stem, coconut oil, and industrial chemicals is a promising area of research that combines sustainability with performance.

This project successfully explored the optimization, and implementation of an organic detergent production process, highlighting the advantages of using environmentally friendly ingredients over conventional chemical-based alternatives. By utilizing plant-based oils and biodegradable additives, the project demonstrated that it is not only feasible to produce effective detergents with minimal environmental impact but also economically viable for large-scale production. The experimental phases confirmed that the organic detergent provided comparable cleaning performance to traditional synthetic detergents.

In conclusion, the project not only provides a practical approach to the production of organic detergents but also contributes to the growing field of sustainable engineering practices. Future work can focus on further refining the formulation, optimizing production efficiency, and expanding the application of organic detergents to other cleaning industries

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