International Journal of Multidisciplinary Research and Publications
ISSN (Online): 2581-6187

Performance of Activated vs. Non-Activated Pineapple (*Ananas comosus*) Waste Adsorbents on Surfactants and pH in Laundry Wastewater

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Abstract**—*****This study evaluated the performance of non-activated pineapple adsorbent (NPA) and citric acid–activated pineapple adsorbent (APA) as low-cost materials for surfactant removal from real laundry wastewater collected in Zamboanga City, where effluents often exceed the limits set by DENR DAO 2016-08. Pineapple wastes (peels, crowns, and cores) were processed into NPA, while APA was chemically modified using 0.5 M citric acid. Batch adsorption tests were conducted at dosages of 5 g/L, 15 g/L, and 30 g/L over a 24-hour contact time. Surfactant concentrations were measured using the Methylene Blue Active Substances (MBAS) method, and pH levels were monitored before and after treatment. The raw wastewater exhibited a surfactant concentration of 119.45 mg/L and a pH of 9.81. NPA-treated effluents showed surfactant concentrations ranging from 102.32 to 380.70 mg/L, with negative removal efficiencies averaging –50.66%, –47.47%, and –138.90% for 5 g/L, 15 g/L, and 30 g/L, respectively, and pH values remaining alkaline, ranging from 8.09 to 10.07. Similarly, APA-treated samples had surfactant concentrations between 123.74 and 447.31 mg/L, with removal efficiencies of –67.19%, –92.95%, and –180.46% for the same dosages, although pH decreased to 5.58–8.36. Neither adsorbent achieved the regulatory limit of 15 mg/L. Neither adsorbent met the 15 mg/L regulatory limit, indicating ineffective and inconsistent performance, likely due to micelle formation, co-contaminant competition, and interference from complex wastewater constituents. Results suggest that while pineapple waste is sustainable, NPA and APA are unsuitable as standalone treatments. Future work should focus on broader wastewater testing, dosage optimization, contact time variation, alternative activation methods, mechanism-focused analysis, and improved experimental cleanliness to improve efficiency and achieve compliance.*

Keywords**—** *Adsorption: Agricultural waste: Citric acid activation: Laundry wastewater: Pineapple waste adsorbent: Surfactant removal.*

I. Introduction

Laundry wastewater is a major but often underregulated greywater stream in developing regions. It typically contains surfactants, builders, bleaches, dyes, and suspended solids that negatively affect water quality when discharged untreated. Among these pollutants, anionic surfactants such as linear alkylbenzene sulfonates (LAS) are of particular concern because of their persistence in aquatic environments, ability to cause foaming, reduction of oxygen transfer, and potential to produce acute and chronic toxic effects on aquatic organisms (Arora et al., 2022; Badmus et al., 2021). In the Philippines, the Philippine Clean Water Act of 2004 (RA 9275) and the Department of Environment and Natural Resources (DENR) Administrative Order 2016-08 establish effluent standards for surfactants, measured as Methylene Blue Active Substances (MBAS), and pH to protect receiving water bodies. However, previous studies have shown that laundry wastewater frequently exceeds these regulatory limits, exhibiting elevated surfactant concentrations, high turbidity, and variable pH values (Jayanto & Widyastuti, 2021).

The increasing generation of laundry wastewater is particularly evident in rapidly urbanizing areas such as Zamboanga City, Philippines, where commercial laundry services have expanded considerably in recent years. Records obtained from the Zamboanga City Business Permits and Licensing Office (BPLO) indicate that the number of registered laundry establishments increased from 125 in 2021 to 178 in 2024, with 154 establishments already recorded as of August 2025. This growth corresponds to increased volumes of wastewater that may enter drainage systems with little or no treatment. Consequently, surfactant-rich effluents can contribute to persistent foaming, reduced dissolved oxygen levels, and the accumulation of detergent residues in aquatic environments, posing risks to both ecosystem and water quality (Arora et al., 2022; Badmus et al., 2021).

To address these concerns, recent studies have explored low-cost and sustainable treatment alternatives based on agricultural waste-derived adsorbents. Lignocellulosic biomasses contain hydroxyl, carboxyl, and phenolic functional groups that facilitate the adsorption of various pollutants, including organic compounds, dyes, and metals (Gupta & Suhas, 2009). Pineapple (Ananas comosus) waste is particularly promising because it is abundant in tropical regions and contains cellulose, hemicellulose, pectin, and lignin, which provide potential adsorption sites (Fouda-Mbanga & Tywabi-Ngeva, 2022). Furthermore, chemical modification through citric acid activation has been reported to improve adsorption performance by introducing additional carboxyl groups, increasing surface acidity, and enhancing porosity, thereby increasing pollutant uptake compared with untreated biomass (Rehman et al., 2024).

Despite the growing interest in agricultural waste-based adsorbents, studies evaluating the effectiveness of pineapple waste for surfactant removal from real laundry wastewater remain limited. Existing research has largely focused on

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synthetic wastewater or on the removal of other contaminants, while the complex composition of actual laundry effluents, characterized by mixed surfactant systems, competing pollutants, and fluctuating pH conditions, may significantly influence adsorption behavior. Moreover, comparative evaluations between non-activated pineapple adsorbents and citric acid-activated pineapple adsorbents for surfactant removal are scarce. This knowledge gap limits the understanding of whether chemical activation provides a meaningful advantage under realistic treatment conditions.

Therefore, this study investigated the performance of non-activated pineapple adsorbent (NPA) and citric acid-activated pineapple adsorbent (APA) as low-cost materials for the treatment of real laundry wastewater collected from a commercial laundry establishment in Zamboanga City. Specifically, the study evaluated the initial wastewater quality in terms of surfactant concentration and pH, determined the surfactant removal efficiencies of NPA and APA at dosages of 5, 15, and 30 g/L, compared the effectiveness of the two adsorbents, assessed changes in pH following treatment, and evaluated the treated effluent against the standards prescribed by DENR Administrative Order 2016-08. Conducted as a laboratory-scale comparative experiment, the study focused exclusively on surfactant and pH parameters using the MBAS method and pH analysis. The findings contribute empirical data on the practical performance of pineapple-based adsorbents under real wastewater conditions and provide insights into their potential application as sustainable, low-cost treatment alternatives for small and medium-scale laundry establishments while supporting the valorization of agricultural residues within a circular economy framework.

II. METHODOLOGY

2.1 Research Design

This study employed a laboratory-scale experimental design to evaluate the adsorption performance of non-activated pineapple adsorbent (NPA) and citric acid-activated pineapple adsorbent (APA) for surfactant removal from real laundry wastewater. Batch adsorption experiments were conducted using three adsorbent dosages (5, 15, and 30 g/L) under identical treatment conditions. Each treatment was performed in triplicate to ensure reproducibility and allow statistical comparison between adsorbents.

2.2 Wastewater Collection

Laundry wastewater was collected from a commercial laundry establishment located along Gov. Alvarez Avenue, Zamboanga City, Philippines. The facility uses commonly available laundry products, including detergent powder, fabric conditioner, fabric cologne, and bleach, making the wastewater representative of typical commercial laundry effluent. Approximately 30 L of wastewater were collected through grab sampling and stored in sealed polyethylene terephthalate (PET) containers. Samples were preserved in an ice chest during transport and analyzed within the recommended holding period.

2.3 Preparation of Adsorbents

Pineapple wastes consisting of peels, crowns, and cores were collected from local fruit vendors and washed thoroughly with distilled water to remove adhering impurities. The biomass was cut into smaller pieces, oven-dried at 110 °C until a moisture content below 10% was achieved, ground, and sieved to obtain particles with a size of ≤250 μm. The resulting material was designated as the non-activated pineapple adsorbent (NPA)

Citric acid-activated pineapple adsorbent (APA) was prepared using NPA as the precursor material. A 0.5 M citric acid solution was prepared and used for impregnation at a solid-to-liquid ratio of 1:10 (w/v). The mixture was soaked for 12 h at room temperature, followed by thermal curing at 140 °C for 2 h to promote esterification and surface modification. The activated material was repeatedly rinsed with distilled water until neutral pH was achieved, oven-dried at 110 °C, ground, sieved to ≤250 μm particle size, and stored in airtight containers until use.

2.4 Microscopic Characterization

Microscopic observations were conducted to qualitatively compare the surface morphology of NPA and APA. Powdered samples were examined using a compound optical microscope under bright-field illumination at 10× magnification. Photomicrographs were obtained to evaluate structural differences resulting from citric acid activation.

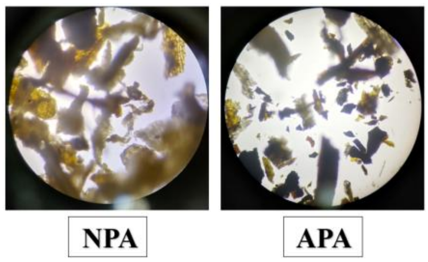

Fig. 1. Microscopic Image of NPA and APA.

2.5 Batch Adsorption Experiments

Batch adsorption experiments were conducted using 1 L wastewater samples placed in individual beakers. Adsorbent dosages of 5, 15, and 30 g/L were added separately for both NPA and APA treatments. The mixtures were manually stirred for 5 min to ensure uniform dispersion and then maintained at room temperature (25 ± 2 °C) for a contact period of 24 h. Light manual mixing was performed every 6 h throughout the treatment period.

After treatment, the mixtures were filtered through Whatman Grade 1 filter paper to separate the adsorbent from the treated wastewater. Filtrates were collected in labeled containers and preserved at ≤6 °C prior to laboratory analysis.

2.6 Analytical Methods

The pH of raw and treated wastewater samples was measured using a calibrated portable multiparameter water quality tester. Calibration was performed using standard buffer solutions at pH 4.01, 6.86, and 9.18 before sample analysis.

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Surfactant concentrations were determined by Chempro Analytical Services Laboratory using the Methylene Blue Active Substances (MBAS) method. Influent and effluent samples were analyzed to determine surfactant removal performance.

2.7 Data Analysis

The data analysis focused on evaluating the treatment performance of non-activated pineapple adsorbent (NPA) and citric acid-activated pineapple adsorbent (APA) in removing surfactants from laundry wastewater and assessing the corresponding changes in pH. Experiments were conducted at adsorbent dosages of 5, 15, and 30 g/L, with each treatment performed in triplicate.

The surfactant removal efficiency was calculated based on the difference between the influent and effluent surfactant concentrations. In the equation, Cinfluent represents the influent surfactant concentration (mg/L), while Ceffluent represents the effluent surfactant concentration (mg/L) after treatment. The percentage removal efficiency was determined using the formula:

$$\%\,\boldsymbol{Removal} = \frac{\boldsymbol{C_{influent}} - \boldsymbol{C_{effluent}}}{\boldsymbol{C_{influent}}} \times \mathbf{100} \tag{1}$$

Descriptive statistics, including mean values and standard deviations, were used to summarize surfactant removal efficiencies and pH measurements. A paired t-test was performed to determine whether significant differences existed between the performance of NPA and APA at a significance level of $\alpha = 0.05$. The resulting effluent quality was further compared with the requirements of DENR Administrative Order No. 2016-08, which specifies effluent limits of 15 mg/L for surfactants (as MBAS) and a pH range of 6.0–9.5.

III. Result and Discussion

3.1 Characterization of Raw Laundry Wastewater

The raw laundry wastewater exhibited an initial MBAS concentration of 119.45 mg/L and an initial pH of 9.81. These values served as the baseline for evaluating the performance of the non-activated pineapple adsorbent (NPA) and citric acid-activated pineapple adsorbent (APA). The measured MBAS concentration exceeded the DENR DAO 2016-08 effluent standard of 15 mg/L by approximately eight times, indicating a substantial surfactant load characteristic of commercial laundry wastewater. Likewise, the influent pH slightly exceeded the allowable range of 6.0–9.5, reflecting the strongly alkaline nature of detergent-rich effluents.

Reported surfactant concentrations in laundry wastewater vary considerably depending on detergent usage, operational scale, and water consumption. The measured concentration of 119.45 mg/L falls within the ranges reported by Braga and Varesche (2014) and González et al. (2025), confirming that the wastewater used in this study represents a moderately to highly contaminated laundry effluent. Similarly, the measured pH is consistent with values commonly reported for laundry wastewater due to the presence of alkaline detergent builders such as sodium carbonate and sodium silicate (Procházková et al., 2022; Huillca-Mamani et al., 2024).

3.2 Surfactant (MBAS) Removal Performance

The MBAS concentrations obtained after treatment with NPA and APA are presented in Table I, while Figure 2 illustrate the corresponding concentration trends.

TABLE I. Surfactant Concentration (MBAS) for NPA and APA.

Adsorbents	Dosage (g/L)	MBAS Concentration Range (mg/L)	Average Removal Efficiency (%)
Influent		119.45	
NPA	5	150.38–219.85	-50.66
	15	158.23–210.81	-47.47
	30	102.32–380.70	-138.90
APA	5	161.80–239.83	-67.19
	15	123.74–330.26	-92.95
	30	137.53–447.31	-180.46

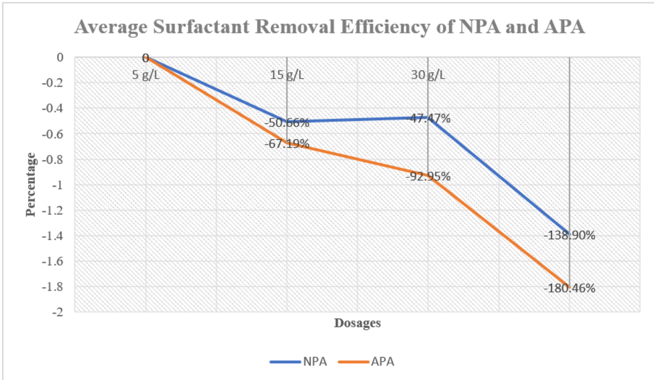

Fig. 2. MBAS Trend for NPA.

For NPA, effluent MBAS concentrations ranged from 102.32 mg/L to 380.70 mg/L. The lowest concentration was observed during the first trial at 30 g/L, corresponding to a removal efficiency of 14.34%. However, this reduction was not reproduced in the succeeding trials, which yielded concentrations of 380.70 mg/L and 338.83 mg/L. Consequently, average removal efficiencies for NPA were −50.66%, −47.47%, and −138.90% at 5, 15, and 30 g/L, respectively.

Similarly, APA did not achieve effective surfactant removal. Effluent MBAS concentrations ranged from 123.74 mg/L to 447.31 mg/L, resulting in average removal efficiencies of −67.19%, −92.95%, and −180.46% at 5, 15, and 30 g/L, respectively. The highest concentration was recorded at the 30 g/L dosage, where the second trial produced an effluent MBAS concentration of 447.31 mg/L.

The predominance of negative removal efficiencies indicates that both adsorbents increased the measured MBAS concentration rather than reducing it. This behavior is likely associated with the release of dissolved organic matter from the pineapple biomass. Pineapple residues contain cellulose, hemicellulose, lignin, pectin, sugars, and other soluble compounds that may leach into solution during treatment. Under alkaline conditions, many of these compounds become increasingly soluble and can interact with methylene blue during MBAS analysis, contributing to elevated apparent surfactant concentrations.

The phenomenon appeared more pronounced for APA than for NPA. Citric acid activation introduces additional carboxyl and carbonyl functional groups to the adsorbent surface, increasing the likelihood of releasing low-molecular-weight acidic compounds during treatment. These compounds may contribute directly to MBAS measurements and explain the more negative removal efficiencies observed for APA. Furthermore, the negatively charged surface generated through citric acid activation may create electrostatic repulsion between the adsorbent and anionic surfactants, thereby reducing adsorption affinity.

The complex composition of real laundry wastewater likely further limited adsorption performance. In addition to anionic surfactants, the wastewater contained fabric softeners, bleaching agents, oils, dyes, fragrances, polymers, and suspended solids. These constituents compete for adsorption sites and alter adsorption equilibria, reducing the effectiveness of both adsorbents. Consequently, neither NPA nor APA demonstrated reliable surfactant removal under the conditions investigated.

To determine whether the observed differences between NPA and APA were statistically significant, a paired t-test was performed using the average removal efficiencies at each dosage level. The analysis indicated that the mean removal efficiency of NPA (−79.01 ± 51.90%) was not significantly different from that of APA (−113.53 ± 59.40%), $t(2) = -3.81$, $p = 0.063$. Since the p-value exceeded the significance level of 0.05, the null hypothesis was not rejected. Although APA exhibited more negative average removal efficiencies, the difference between the two adsorbents was not statistically significant.

Fig. 3. Paired t-test Distribution (df = 2).

The Shapiro–Wilk test further indicated no significant departure from normality ($p = 0.409$). However, the statistical power of the analysis was limited by the small sample size. Although a large effect size (Cohen's d = 2.20) was obtained, the low statistical power (0.08) suggests that additional observations would be required to confidently establish whether a true difference exists between the two adsorbents.

3.3 pH Modification Performance

The pH values obtained after treatment with NPA and APA are presented in Tables II.

Treatment with NPA resulted in average pH values of 8.98, 8.76, and 9.33 at 5, 15, and 30 g/L, respectively. Although these averages remained within the DAO 2016-08 allowable range, individual trials showed noticeable variability. The highest recorded value was 10.07 during Trial 1 at 5 g/L, exceeding the regulatory limit. This increase may be attributed to the release of alkaline minerals and soluble ash naturally present in untreated pineapple biomass.

TABLE II. NPA and APA pH Values of the Influent and the Effluent Samples.

Adsorbents	Dosage (g/L)	pH Range
Influent		9.81
NPA	5	8.27–10.07
	15	8.09–9.88
	30	9.10–9.78
APA	5	7.25–8.36
	15	6.60–7.25
	30	5.58–6.89

In contrast, APA produced a more consistent reduction in pH. Average pH values decreased from 7.97 at 5 g/L to 6.99 at 15 g/L and 6.39 at 30 g/L. The observed reduction reflects the introduction of acidic functional groups during citric acid activation and the possible release of residual citric acid species into solution. These acidic groups provide hydrogen ions that neutralize alkalinity and shift the wastewater toward neutral conditions.

The pH results demonstrate that citric acid activation significantly enhanced the ability of the adsorbent to regulate alkalinity. Unlike NPA, APA consistently reduced pH across all dosage levels and generally maintained the treated wastewater within the regulatory range.

IV. CONCLUSION

This study evaluated the performance of non-activated pineapple adsorbent (NPA) and citric acid-activated pineapple adsorbent (APA) for the treatment of real laundry wastewater containing elevated concentrations of anionic surfactants. Initial characterization of the wastewater revealed a MBAS concentration of 119.45 mg/L and a pH of 9.81, indicating a moderately to highly contaminated alkaline effluent that exceeded the DENR DAO 2016-08 effluent standards for surfactants and slightly exceeded the allowable pH range. These values are consistent with those commonly reported for commercial laundry wastewater and reflect the presence of detergent-derived surfactants and alkaline builders.

The results demonstrated that neither NPA nor APA effectively removed surfactants from the wastewater. Across all dosage levels (5, 15, and 30 g/L), the treated effluents generally exhibited MBAS concentrations higher than the influent, resulting in predominantly negative removal efficiencies. NPA produced average removal efficiencies of −50.66%, −47.47%, and −138.90% at 5, 15, and 30 g/L, respectively, while APA yielded average efficiencies of −67.19%, −92.95%, and −180.46%. These findings indicate that both adsorbents failed to achieve meaningful adsorption of anionic surfactants under actual wastewater conditions. The observed increases in MBAS concentrations suggest that dissolved organic compounds released from the pineapple biomass, together with wastewater matrix interactions and analytical interferences associated with the MBAS method, contributed to elevated apparent surfactant concentrations following treatment.

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Although citric acid activation altered the physical and chemical characteristics of the pineapple biomass, no improvement in surfactant removal performance was observed. Microscopic examination revealed increased surface roughness and fragmentation in APA compared with NPA; however, no substantial pore development was evident. Statistical analysis further confirmed that the difference in surfactant removal performance between NPA and APA was not significant ($p = 0.063$), indicating that citric acid activation did not provide a measurable advantage under the experimental conditions employed. These findings suggest that surface modification through citric acid activation alone is insufficient to overcome the challenges associated with treating complex laundry wastewater containing mixed surfactants, organic matter, and competing contaminants.

In contrast to surfactant removal, both adsorbents influenced the pH of the treated wastewater. NPA generally maintained alkaline conditions, producing pH values between 8.09 and 10.07, whereas APA consistently reduced pH to values ranging from 5.58 to 8.36 due to the introduction of acidic functional groups during activation. While APA demonstrated greater capability for pH adjustment and produced effluent pH values that were generally closer to the regulatory range, this improvement did not translate into enhanced surfactant removal. The results therefore indicate that pH modification alone is not a reliable indicator of adsorption effectiveness in surfactant-rich wastewater systems.

Overall, neither NPA nor APA achieved compliance with the DENR DAO 2016-08 surfactant limit of 15 mg/L, and both adsorbents exhibited limited reliability as standalone treatment materials for real laundry wastewater. The findings demonstrate that pineapple-based adsorbents, whether untreated or citric acid-activated, are inadequate for effective removal of anionic surfactants in complex wastewater matrices. Consequently, additional adsorbent modification strategies, advanced activation techniques, or integration with complementary treatment processes will be necessary to enhance treatment performance and achieve regulatory compliance for surfactant-rich laundry effluents.

APPENDIX

Fig. 4. Collected Pineapple Wastes from Local Vendors.

Fig. 5. Oven-drying of the Biomass Samples.

Fig. 6. Stored NPA Powder.

Fig. 7. Stored APA Powder.

Fig. 8. NPA and APA Adsorption from Three Trials.

ACKNOWLEDGMENT

The researcher expresses sincere gratitude to the individuals whose support and guidance made this study possible. Foremost, the researcher extends the utmost respect and appreciation to the Examination Committee Chairman, Dr. Lizamyl R. Laping, for her valuable insights and constructive comments throughout the development of the research. Deep appreciation is likewise extended to the panel members, Engr. Dwinsel Maramba and Engr. Jeshier John H. Feliciano, for their critical evaluations, meaningful suggestions, and professional guidance that greatly strengthened the study.

Heartfelt thanks are also given to the researcher's thesis adviser, Engr. Ilde B. Deloria, whose expertise, patience, and unwavering support were instrumental in completing the experimental procedures and understanding the technical aspects of the study. His mentorship has had a profound impact on the quality and direction of this research.

The researcher sincerely acknowledges Mrs. Ferilyn Rodriguez, Manager of Luxe Café Laundry, for granting permission to collect wastewater samples essential for the experimental analysis. Her cooperation and assistance played an important role in facilitating the data collection process.

The deepest appreciation is extended to the researcher's parents, Mr. Franklin O. Galabin and Mrs. Rosie L. Galabin, for their steadfast moral support, financial assistance, and constant encouragement. Their sacrifices and belief in the researcher's capabilities have been a continuous source of strength. Special thanks are also given to the researcher's sibling, Ms. Irah Mae L. Galabin, for her unwavering moral support and motivation, as well as to the researcher's classmates for their solidarity and encouragement throughout the research journey. With gratitude, the researcher acknowledges that the successful completion of this thesis would not have been possible without the collective support of all these individuals.

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