INVESTIGATION OF SURFACANT SURFACE TENSION AND ITS CORRELATION WITH TEMPERATURE AND CONCENTRATION

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1 Abstract
Surface tension is a property that defines the energy per unit area of a liquid-gas interface. The purpose of this study is to investigate the surface properties and the process of aggregation of surfactant micelles in detergents. By using a drop-weight method, the surface tension of detergent solutions was determined under standard pressure condition 1 bar. A 100% concentrated detergent solution was subject to heating. Data analysis showed that surface tension decreased linearly with increasing temperature, as represented by the equation $\gamma = -0.1766 \text{ mN/(m} \cdot \text{K})(x) + 94.654 \text{ mN/m}$, where $x$ is temperature. When subjected to variations in concentration, surface tension of the detergent decreased until reaching the critical micelle concentration, as modelled by the segmented linear regression $\gamma = -0.565 \text{ mN/(m} \cdot \%)(x) + 61.7 \text{ mN/m}$ when $x < 50\%$, and $\gamma = 33.45 \text{ mN/m}$ when $x \geq 50\%$, where $x$ is concentration.

2 Introduction
Surface tension of a solvent or solution, denoted by the Greek letter gamma $\gamma$, represents a well-defined physico-chemical property. It is caused by cohesion of fluid molecules and allows for its resistance of an external force \cite{1}. The unit of measurement has dimensions of force per unit length, or equivalently, energy per unit area. Molecules at the surface experience a net inward force which results in internal pressure. Considering the isoperimetric inequality, liquid surfaces minimize its energy of configuration by contracting to minimal area \cite{2}. Surface tension is ubiquitous in the natural world. For instance, water striders are able to float on top of water due to the surface tension of water providing the restoring force to the insect's weight. In surface and colloidal chemistry, surface tension can be useful in characterizing the chemical activity, adsorption, and dissolution of materials \cite{3}.

A specific branch of study in surface chemistry is the surface tension of surfactants, which are surface-active agents that adsorb to a fluid surface. Besides the common usage of surfactants in food additives, detergents, and cosmetics, these molecules are often used as surface active agents in the production of drugs within both medicinal and pharmaceutical sciences \cite{4}. When dissolved in solvent, surfactants disrupt the intermolecular interactions of the liquid, thereby decreasing the solvent's surface tension. Water-soluble polymers have amphipathic structures, with non-polar hydrophobic parts and hydrophilic parts that favour interaction with polar solvents such as water \cite{3}. The formation of micelles can be understood through principles of thermodynamics; micelles can form spontaneously because of a balance between entropy and enthalpy. In water, hydrophobic effects and London van der Waals forces between water and surfactant molecules drive the micellization process.

In the present study, we have selected a particular detergent brand named “Palmolive Original.” The primary surfactant agent is lauramidopropylamine oxide, a non-ionic surfactant. As its name suggests, non-ionic surfactants do not possess electrical charges \cite{9}. The substance's surface properties were characterized by measuring the surface tension of the detergent at different temperatures and concentrations. The du Nuöy ring method was adopted as a model for the conduction of surface tension measurement \cite{5}.

3 Method
3.1 Equipment Calibration
The main component of the apparatus is the dial-o-gram (Ohaus, #1650-WO). Figure 1 depicts the setup of our experiment. A stiff copper wire of negligible mass was used to construct a ring and secured to the beam. To ensure the copper ring was circular and completely flat, the wire was tied around a cylindrical container and hammered into circular conformation. The ring was flattened using a hammer on a smooth table. The ring's flatness and circular shape were crucial to achieving even distribution of the pulling force upon the liquid surface. When securing the extended arm of the ring onto the beam, tweezers were used to minimize the wiggle room of the twisted wire. A 1 kg calibration weight was placed on the stainless steel plate and weight was adjusted on the beam to create an initial rough balance. A clean and lightweight Styrofoam cup was placed onto the plate. The fine-tuning knob was adjusted so the beam hit the zero-mark, which indicated the exact point of balance. This marked the initial position.
3.2 Measurement of Surface Tension
A 250 mL beaker was used to hold the solution subjected to measurement. The beaker was filled to precisely the 200 mL mark. This mark was the level that allowed the ring to rest on top of the liquid surface without disrupting the equilibrium position of the beam. The cup was pre-weighed on an analytical balance to obtain the mass to 5 significant digits. Since surface tension is considered a weak force compared to a gravitational pull by the applied force, a dropper bottle was used to add water drops to the Styrofoam cup in small increments until the beam at the delicately balanced position was tipped and the ring detached from the liquid surface. Clean water from the tap was used. The cup and added water were weighed on the analytical balance, and the mass of water added was calculated. Five replicates were carried out for each trial. Note the dial-o-gram was used because it has a high degree of sensitivity to minuscule additions of mass. An addition of one hundredths of a drop of water can be sufficient to tip the balance.

3.3 Mathematical Derivation of Surface Tension Formula
Considering the du Noüy formula \[5\], the surface tension is given by \[\gamma = \frac{Mg}{2L}\] where \(\gamma\) is the surface tension, \(M\) is the mass used to applied force, \(g\) is the gravitational acceleration (9.807 m/s\(^2\) was used throughout the experiment), and \(L\) is the mean circumference of the ring. The unit of measurement is force per unit length with SI units N/m. The surface tension of the liquid was calculated using principles of torque and rotational dynamics \[5\]. Torque is the product of arm length \(r\) with the force component \(F\) that is perpendicular to the radial line. The formula is given by \(\tau = rF\sin\theta\), where \(\theta\) is the angle between the moment arm and the force applied. In the experiment, \(\theta = 90^\circ\), since the surface tension and the weight of water drops were both tangent to the beam. Initially, the system was at rotational equilibrium. As shown in Figure 2, the addition of drops of water to the Styrofoam cup resulted in a gravitational torque greater than the torque exerted by surface tension. This caused the system to rotate counter-clockwise about the fulcrum (the pivot point). Given \(r_R\) is depicted as the torque on the ring side where tension exerts force and \(r_P\) as the torque on the plate side where mass exerts a downward force, then the formula for surface tension can be derived as follows:

\[
(F_{\text{tension}})(r_R) = (F_{\text{mass}})(r_P)\\
\gamma 2 L r_R = M g r_P\\
\gamma = \frac{M g r_P}{2 L r_R} .
\]

\[1\]
3.4 Statistical Analysis
The uncertainty ($\sigma_m$) in each measurement of surface tension was derived from standard deviation. The formula is $\sigma_m = \sigma / \sqrt{N}$, where the standard deviation is calculated as follows:

$$\sigma = \sqrt{\frac{1}{N-1} \sum_{i=1}^{N} (y_i - \mu)^2},$$

(2)

where the mean ($\mu$) is

$$\mu = \frac{1}{N} \sum_{i=1}^{N} y_i,$$

(3)

For each linear regression model in the form of $y = mx + b$, the best-fit line was generated on Excel 2010 by using the weighted best-fit slope, its uncertainty $\delta m$ and $y$-intercept $b$ as displayed below:

$$m = \frac{1}{\Delta} \left[ \sum_{i=1}^{N} \frac{1}{(\delta y_i)^2} \sum_{i=1}^{N} x_i y_i - \frac{1}{N} \sum_{i=1}^{N} x_i \sum_{i=1}^{N} y_i \right],$$

(4)

$$\delta m = \sqrt{\frac{1}{\Delta^2} \sum_{i=1}^{N} \frac{1}{(\delta y_i)^2}},$$

(5)

$$b = \frac{1}{\Delta} \left[ \sum_{i=1}^{N} \frac{x_i^2}{(\delta y_i)^2} \sum_{i=1}^{N} y_i - \frac{1}{N} \sum_{i=1}^{N} x_i \sum_{i=1}^{N} y_i \right],$$

(6)

where

$$\Delta = \sum_{i=1}^{N} \frac{1}{(\delta y_i)^2} \left[ \sum_{i=1}^{N} \frac{x_i^2}{(\delta y_i)^2} - \left( \frac{\sum_{i=1}^{N} x_i}{\sum_{i=1}^{N} (\delta y_i)^2} \right)^2 \right].$$

(7)

Each resultant linear regression model is then evaluated using weighted least square analysis derived as follows:

$$Z^2 = \frac{1}{N} \sum_{i=1}^{N} \left( y_i - f(x_i) \right)^2,$$

(8)

3.5 Varying Temperature
The temperature of a 100% concentration surfactant solution and of clean tap water was varied. Identical procedures of temperature variations were carried out for both types of solutions. A 250mL beaker was filled with 200mL of solution and put in a standard refrigerator to be cooled to 273K. A thermometer was used to monitor the temperature drop. Then the beaker was taken out and placed on an insulating mat to prevent heat to be transferred to or from the solution to the surroundings. Surface tension was measured using standard protocols as delineated in the above procedural section. The solution was heated in 5K increments with the glass beaker placed in a pan. The pan is then placed on an electric stove to prevent the glass from cracking due to large pressure changes from cooling or heating. The temperature was monitored using a thermo-anemometer (Extech Instruments), which detects air velocity, relative humidity and temperature of the system. This instrument was used to ensure the uniformity of environmental conditions other than experimental variable, which is temperature.
3.6 Varying Concentrations
Surface tension was also correlated with surfactant concentration. “Palmolive Original” detergent was used as the surfactant. Multiple concentrations of detergent were employed to ensure accuracy of the direct relation between surfactant and change in magnitude of surface tension. Varying concentrations without affecting volume was achieved by the volume replacement method, where the volume was set to 200mL. The following equation was used for concentration and dilution, respectively:

\[
\Delta V(x_k, x_{k+1}) = \frac{200(x_{k+1} - x_k)}{1 - x_k}
\]

\[
\Delta V(x_k, x'_{k+1}) = \frac{200(x_k - x'_{k+1})}{x_k}
\]

where \(\Delta V\) is the volume replaced to achieve a certain concentrated or diluted concentration. This volume of solution was either discarded and replaced with pure detergent when increasing surfactant concentration or replaced with water when performing dilutions. The calculated amount should result in a mixture of desired concentration while returning its volume to 200mL as the control volume. \(x_k\) is the initial concentration, \(x_{k+1}\) is the increased concentration, and \(x'_{k+1}\) is the decreased concentration upon dilution. Concentrating from 0% detergent to 100% detergent and its reverse (i.e., diluting) were carried out to optimize the precision of each concentration data point. This resulted in the number of replicates doubling to ten.

4 Results and Analysis
4.1 Temperature Dependence of Surface Tension

Linear relation is observed and hypothesized for both Figure 3 and Figure 4 using \(y = mx + b\), where \(m\) is the slope and \(b\) is the y-intercept of the model function. Using the weighted least square fitting as the linear model, it was determined that for water, the linear model yields a minimum weighted chi-square value of 8.361, with \(m = -0.288 \text{ mN/m} \cdot \text{K}\) and \(b = 152.86 \text{ mN/m}\), resulting in a critical temperature of 531.024K. The critical temperature marks the point at which surface tension is expected to drop to zero (equivalent to the x-intercept). However, the extrapolated value of the critical temperature for water has no physically relevant meaning because water would already be in gas phase at 373K. With the same method, it was determined that for detergent, the linear model yields a minimum weighted chi-square value of 2.310 with \(m = -0.177 \text{ mN/m} \cdot \text{K}\) and \(b = 94.654 \text{ mN/m}\), resulting in a critical temperature of 535.896K. The linear model for water shows a steeper slope and a higher y-intercept than the parameters of the linear model for detergent.
Distinct discretization of surface tension decrement can be observed in Figure 3 and Figure 4. Consecutive differences are not uniform. Significant drops in value occur in a stepwise fashion. The cause was first hypothesized to be the discrete mass added for each drop of water and the sensitivity of the equipment used. To test the hypothesis, the weight of several drops of water was determined and was divided by the number of drops to obtain mass per drop. Mean mass is applied to equation (2) to obtain the mean gravitational force per length per drop. This renders the value 0.212 mN/m per drop, which was compared to mean significant changes in surface tension in Figure 3 and Figure 4. Mean significant changes are quantified as the mean of the greatest ten consecutive fluctuations of data. In Figure 3, the mean significant change in surface tension is 4.422 mN/m. In Figure 4, the mean significant change in surface tension is 2.122 mN/m. Both results show low correlations to the mean gravitational force exerted per drop. The higher values in fluctuation can be rationalized with the additional applied force when squirting the water into the Styrofoam cup.

4.2 Concentration Dependence of Surface Tension

![Figure 5: Surface Tension of Water and Detergent at Various Temperatures at Standard Pressure Condition 1 bar. Bars represent standard error calculated according to the appendix. Each data point represents a total number of five replicates (n = 5).](image)

![Figure 6: Surface Tension of Water-Detergent Mixture Measured with Different Concentrations at Room Temperature 298K and Standard Conditions with Varying Concentrations. Bars represent standard errors. Each data point represents a total number of ten replicates (n = 10). $\chi^2 = 20.217$, $m = -0.565 \text{ mN/m} \cdot \text{K}$, $b = 61.700 \text{ mN/m}$, $C = 50\%$.](image)

Considering the formula for uncertainty shown in the procedural section, the surface tension of water at pressure 1 bar and room temperature (273K) was measured to be 70.000 ± 3.200 mN/m and the surface tension of detergent is 36.000 ± 1.700 mN/m. By comparing Figures 3 and Figure 4 in the composite graph Figure 5, the surface tension of water is consistently higher than the detergent, by nearly 50%. There is no ambiguity with this statement since the uncertainty ranges do not overlap. However, as shown by the small error bars, this absence of ambiguity may be due to underestimation of error propagation in measurements or instrumental error (Please refer to Qualitative Error Analysis for in-depth discussion).

In Figure 6, the surface tension after the critical point decreases steadily with increasing surfactant concentration. However, close inspection reveals that the decreasing nature halts at about 50% concentration. Surface tension after this concentration lacks a particular trend; it could be oscillating, increasing, or constant. Most of the data points demonstrate significance with no overlaps. Minimum uncertainty is observed at 50% concentration and maximum uncertainty is determined to be at 62.500%. Hence, linear regression termed the “hockey stick function” was used to fit the data shown in Figure 6:

$$y = \begin{cases} m_1x + b_1, & x < C \\ m_2x + b_2, & x \geq C \end{cases}$$

where $m_1$ and $m_2$ are slopes, $b_1$ and $b_2$ are y-intercepts, and C is the critical breakpoint of the segmented linear regression where behaviour changes dramatically. The parameters are generated by determining the least chi-square value. For this segmented linear regression model, since the best-fit slope and y-intercept cannot be determined with the
aforementioned formula, manual minimization of fit parameters was employed. The minimality of the slopes, y-intercepts and value of C were confirmed by plotting trial values to generate parabolic functions. The point at which the derivative is zero depicts the minimum value of the parameter. The set of graphs are shown in Figure 7 below.

![Graphs showing chi-square values](image)

**Figure 7. Manual Minimization of Chi-Square Values.** The parameters were generated by determining the minimum points on the graphs, each corresponding to a specific parameter. As shown in Figure 6, the values are $\chi^2 = 20.217$, $m = -0.565 \text{ mN/m} \cdot \%, b = 61.7 \text{ mN/m}$, $C = 50\%$.

The uncertainty associated with each data value in Figure 6 is one sigma bar, which suggests two-thirds of the data values should cross the fit line. However, only two-ninths of the error bars cross the line. This may be due to the aforementioned underestimation of error.

5 Discussion

5.1 Surface Activity of Surfactant

The cause of the general decreasing trend in the surface tension of detergent-water mixture may be due to the presence of the cleaning and foaming agent lauramidopropylamine oxide (C17H36N2O2). This molecule is a nitrogenated, non-ionic surfactant [9]. As shown in Figure 8, non-ionic surfactants contain tertiary amine oxides that are often used as emulsifiers since they have high solubility in water [9]. Lauramidopropylamine oxide contains a long hydrophobic alkyl chain and a highly polar N-O bond. The nitrogen atom involved in this bond readily donates its electrons, resulting in a negative oxygen atom which readily captures a proton in aqueous solution [7]. The highly polar N-O bond drives the hydrophilic interactions with water, subsequently modifying and disrupting the hydrogen bonding between water molecules. The amphiphilic property of the surfactant molecule also allows this hydrophilic end to adsorb to the fluid surface. The collective energy that generates water's high surface tension decreases.

![Chemical structure of lauramidopropylamine oxide](image)

**Figure 8. Chemical structure of lauramidopropylamine oxide.** The highly polar N-O bond in lauramidopropylamine oxide disrupts the hydrogen bonding between water molecules, causing surface tension of the aqueous solution to decrease linearly until reaching the critical micelle concentration.

5.2 Critical Micelle Concentration

The position upon which the plateau is reached in Figure 6 can be attributed to the critical micelle concentration (CMC). In colloidal and surface chemistry, CMC is defined as the concentration of surfactant above which micelles spontaneously form in solution [1][7]. The value of CMC and surface tension itself are intensive thermodynamic parameters [4]. These quantities are therefore dependent upon temperature, pressure, and on the presence and concentration of other surface-active electrolytes [8]. The value of the critical micelle concentration quantifies the degree of packing and the orientation of the adsorbed surfactant molecules [1]. As surfactant concentration increases, surface coverage of surfactant molecules increase, and the maximum coverage are reached at CMC [1][9]. At that point, molecules begin to aggregate into micelles. Monomers and micelles thereafter exist in dynamic equilibrium, and the spacing between adjacent molecules attains its most favourable distance to minimize the intermolecular potential energy. Any subsequent formation of micelles will have minor effects on surface tension.

The surface tension of the surfactant at which the experimental CMC value was reached was compared to those attained by Atta et al. [1] who worked with non-ionic surfactants under similar conditions. Shown in Figure 6, the critical micelle concentration was determined to be 50% surfactant concentration. The average constant surface tension
was 33.45mN/m at 298K based on the segmented linear regression model. Atta et al. [1] showed that the non-ionic surfactant CMC was reached at surface tension 34.8 ± 0.5mN/m at 303K.

When comparing Figure 3 and Figure 4, the detergent surface tension decreased much more slowly than did water. This is because in the 100% concentrated surfactant solution, the area per molecule at the surface decreased at the surface due to increased dehydration of the hydrophilic group at higher temperature [7] yet increased due to enhanced molecular motion and thermal agitation at higher temperatures. The two opposing forces result in lower decreasing rate for surfactant surface tension when compared to that of water.

5.3 Qualitative Error Analysis

Some transparent soap bubbles formed around the copper ring and between the ring and the surface of the surfactant as the ring was tilted out of the solution. This made it more difficult to distinguish the exact point at which the ring loses contact with the liquid surface. This would result in a slightly greater amount of water being added to the cup than the volume sufficient to break surface tension. As observed in Figure 3, there are large fluctuations from 273K to 308K, and the values do not correspond to the slope of the general decreasing trend of the measurements above 308K. Bubble-formation was frequent in the range 273K to 308K. Large uncertainties were associated with measurements at lower temperature, as can be seen in Figure 3. The maximum error was detected at 288K temperature, with surface tension 43.460 ± 2.460mN/m.

Due to the ring's frequent contact with the detergent, the ring's smooth wax layer was observed to dissolve. The strength of contact between the liquid surface and the wax layer of the ring would be significantly different from the tension between the surfactant surface and the rougher, grey metal layer underneath the wax. The rough layer of the wire would, in principle, have greater interaction with the water as the wax layer is smoother. This would result in the surfactant clinging more tightly to the ring and exert a larger resistant force to the upward pull. The wax layer has a more hydrophobic surface [4][8]. This results in hydrophobic repulsion that increases the fluid's energy per unit of surface area. The fluid would have a greater tendency to minimize its contact with the waxy surface than to the rougher ring surface [6][7]. As can be observed in Figure 3 and Figure 4, the values remain at a generally constant value with smaller fluctuations. The significantly stronger pull exerted by the non-waxy layer of wire may have contributed to similar measurements.

Tap water contains impurities such as electrolytes, sediments and minerals. This would cause the measured surface tension of water to deviate from literature values (see Appendix). The experimental surface tension values are lower than literature values, especially at temperatures above 298K. The greatest deviation from literature value can be seen at 368K: the measured tension was 50.610 ± 1.250mN/m, whereas the value listed in the CRC Handbook was 59.87mN/m. The 9.260 ± 1.250mN/m difference marks a significant deviation. The general decreasing trend in Figure 3 and Figure 4 may be attributed to increased thermal motion and energy of the molecules with increasing temperature. However, the presence of electrolytes disrupts hydrogen bonding in water molecules. Since intermolecular forces were already weakened before heating, the molecules could attain greater degree of freedom, resulting in lower than predicted surface tension.

5.4 Future Directions

Discrepancy in concentration of mixture may be due to measurement of insufficient accuracy. A graduated cylinder with smaller and more accurate increments of measurement should be used in further investigations. The material of the ring should be switched plastic to prevent degradation of the ring’s surface layer. To increase confidence in the uniform sizes of water drops added as weight, a Pasteur pipet should be used.

When detergent solution was heated to 353K, intense bubbling occurred and the detergent was observed to undergo drastic compositional change, resulting in a more opaque and deeper green solution. The cause of this phenomenon was not known. It may be useful to utilize spectroscopy, generating reflection imaging using lasers, to detect the decomposition mechanism of the surfactant surface layer or layers [1][9]. Further studies of this surface tension of the surfactant solution and of water-surfactant mixtures should be conducted via measurement of the fluid's contact angle in a capillary tube [1][9].
6 Conclusion
We have concluded that under standard pressure condition 1 bar and 298K, the surface tension of “Palmolive Original” surfactant solution decreases linearly as temperature increases, as indicated by the weighted least square fit \( y = -0.177 \text{ mN/(m-K)} + 94.654 \text{ mN/m} \), where \( x \) is temperature. When concentration was varied at constant temperature, surface tension decreased linearly until a particular concentration, which is \( C = 50\% \) for the non-ionic surfactant. Beyond that threshold concentration, the surface tension was interpreted to remain constant. The “hockey stick function” was used to model the trend, with the initial linear \( y = -0.565 \text{ mN/(m \%)}(x) + 61.7 \text{ mN/m} \) when \( x < 50\% \), and \( y = 33.45 \text{ mN/m} \) when \( x \geq 50\% \), where \( x \) is concentration. This model adsorption isotherm is extremely useful in characterizing the surface properties of the surfactant solution. The surface tension of water was observed to be consistently higher than the surface tension of surfactants. These model parameters allow us to make reasonable predictions of the mechanisms of micelle-formation and the energy at the liquid-gas and liquid-solid interface.

7 Acknowledgement
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8 Literature Cited

9 Appendix
9.1 Literature Values for Water Surface Tension

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Table 1: Literature Values for Water Surface Tension from CRC Handbook, AIP Handbook, and Lange's Handbook. The values are derived from three published handbooks. Highlighted values demonstrate complete agreement with experimental data range. Since CRC Handbook provides temperatures with increments of 2K, odd number temperatures are estimated by averaging adjacent values with common difference of 1K. 273.15K is estimated to be 273.15K and 371.05K is estimated as 371.15K. The first surface tension for 298.15K is taken from initial measurement of water surface tension at room temperature and standard condition. The bottom value is taken during heating the substance.