Synthesis & Characterization of Glycineand Sarcosine Based Surfactants

Rutu Parikh, Faiz Khan, Amit P Pratap

Abstract— Surfactants are multiutility products and have large application in different fields but primarily they are derived from petroleum non-renewable feedstock. Biocatalysis and renewable raw materials can bring about positive changes from conventional method for production of green surfactants. However, biocatalysis involves the use of enzymatic synthesis, which is not feasible in an industrial setup. Synthesis employing renewable materials is one of the better and viable options. Amino acid based surfactants belong to the class of surfactants with high biodegradability, low toxicity and excellent surface active properties, which make them highly attractive to the cosmetics and personal care industries. Two series of surfactants based on glycine and sarcosine were synthesized with dodecanoic acid (lauric acid), tetradecanoic acid (myristic acid), hexadecanoic acid (palmitic acid) and octadecanoic acid (stearic acid). The reaction conditions and surface properties were studied. Sarcosine based surfactants exhibited exceptional foaming tendency and stability. These studies revealed that sodium N-acyl glycinate and sarcosinate based surfactants can be used in household and skin care formulations.

Index Terms— Amino acid, Amphoteric Surfactant, Biodegradable, Glycine, Sarcosine, Schotten–Baumann synthesis, Surface active properties.

1 INTRODUCTION

Surfactants are among the most widely used productsin many industries like household (61%) followed by industrial processes (25%), personal care (8%) and specialty cleaning (6%). They represent one of the most useful chemicals that constitute an indispensable part in various consumer products. Most of the surfactants in the market are synthesized using a combination of petrochemicals and natural feedstock. Synthetic surfactants such as alkyl phenol ethoxylates though have excellent surface active properties, lack in biodegradability and biocompatibility aspects [1]. Therefore, there has been a growing trend towards the development of surfactants that are ecologically and environmentally friendly with better surface active properties. Surfactants derived from sugar and amino acids are biocompatible because of the low toxicity of the raw materials and quick biodegradability [2]. Surfactants from amino acid are derived from plant based products that include a hydrophilic part, which is obtained by enzymatic synthesis and a hydrophobic part that is obtained from natural oils [3]. If we compare the amino acids based surfactants with other conventional surfactants, we have that the latter have enhanced surface properties, low skin irritation potential, quick biodegradation, and superior antimicrobial & antifungal activities.

The combination of various polar amino acids and nonpolar hydrocarbon chain compounds produces amino acids based surfactants with high surface activity. Amino acid based surfactants represent a high structural similarity to natural surfactants (Eg. lipoamino acids). As a result, they display a higher compatibility and low toxicity and hence, the preferred choice in a host of different pharmaceutical and cosmetics formulations. Due to structural difference in the amino acid, the surfactants produced vary in various physiochemical and biological properties.

Amino acids based surfactant finds several applications in different areas. They were found active again various disease causing bacteria, viruses and tumors [4]. Lipo amino acids based surfactants can be used in pharmaceutical, food, personal care and cosmetic formulations [5] due to their excellent emulsifying and antimicrobial activities. Aromatic amino acids such as tyrosine and phenylalanine based surfactants are well known for drug related activities [6].

Recent research is targeted toward the development of surfactants from renewable sources, with a view to replacing surfactants from non-renewable petroleum feedstock. Amino acid based surfactants were reported as green surfactants which were prepared from amino acid/peptides and fatty acid obtained from renewable vegetable oil sources [7].

Amino acid based surfactants are of great interest because of their low toxicity and high biocompatibility. The hydrophobic chain can be introduced in form of an ester, acyl, alkyl or amide derivative [3]. Ryonsuke and Takashi [8] mixed acid anhydride to react with the amino acids. Though the method is a simple and convenient process for preparing N-higher aliphatic acyl derivatives of amino acid, its subsequent purification is a heavy task to be accomplished. Lipases were used by RaoValivety to synthesize amino acid based surfactants. He prepared simple monoesters to complex bola and Gemini amphiphiles with lipases. [9]. Althouh enzyme sythnesis is a ecofriendly and energy efficient, but Lipase application is seldom used for industrial application due to its low conversion, long reaction time and expensiveness. For mass based production, Schotten-Baumann condensansation is widely used for its simple sythnesis process. [10].

There have been various studies on the surface-active properties amino acids based different surfactants [11] [12]. Ohta et al. [12] prepared six kinds of N-hexadecanoyl amino acid surfactants, and discussed the relationship between the Krafft temperature and the amino acid residue. Mhaskar et al. [11] studied the effect of structural variation in fatty acid and amino acid moieties on the surfactant properties of sodium salts of N-acyl condensates of amino acids. Sreenu et al. [13] investigated the change in the surface active and antimicrobial properties of N-palmitoyl amino acid with respect to different

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amino acids and a subsequent comparison with Sodium lauryl sulfate for surface activity and Neomycin sulfate for antimicrobial activity comparison. Amino acid based surfactants have also been mixed with other surfactants in order to study the aggregate behavour and micelle & vesicle formation in the mixture [14] [15].

This study focuses on the synthesis of glycinates and sarcosinates based surfactants using schotten-baumann[10] condensation method for obtaining mild surfactants. Synthesis of glycinates and sarcosinates based surfactant is optimized for its operational parameters, so as to get higher yields and conversion. comparative studies for surfactant properties like foaming, wetting, emulsion stability, surface tension(SFT), interfacial tension (IFT) and critical micelle concentration (CMC) is carried out so as to understand , use of surfactants for different applications. Surfactants are characterized by FTIR and later its application is done in personal care products to check its efficacy.

2 Materials & Methods

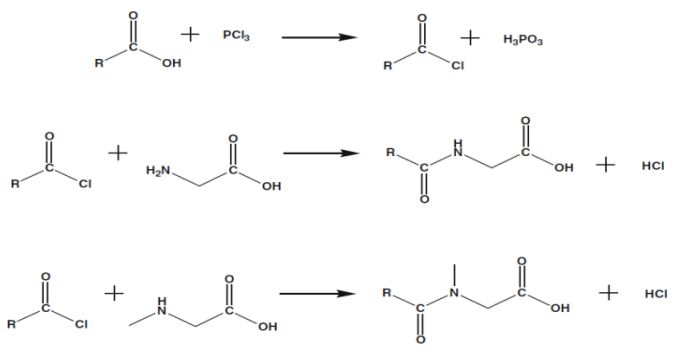
2.1 Material

Dodecanoic acid, tetradecanoic acid hexadecanoicacids were purchased from Godrej Industries, Mumbai (98-99 % pure). Glycine was purchased from Thomas Baker, Mumbai (>99 % pure). Sarcosine was procured from HiMedia, Mumbai (>97.5 % purity). Sodium hydroxide, methanol, concentrated hydrochloric acid were ofanalyticalgrade and procured from Thomas Baker, Mumbai. Phosphorus trichloride was purchased from LobaChemie, Mumbai (>99 % pure). Distilled water was used in all the measurements and experimental procedures.

IR spectra were recorded on Shimazdu (Model No: IR Affinity-1S) FT-IR spectrophotometer.

2.2 Synthesis

The reaction leading to N-acyl amino acid is based on Schotten-Baumann condensation reaction. The hydrophobic entity is attached to the amino group of the amino acid through a reaction with fatty acyl chloride. The process reaction steps are exemplified in the reaction scheme depicted in figure 1. The sodium salts of N-acyl amino acids are generated by neutralizing with a proportional molar quantity of sodium hydroxide.



R=$C_{11}H_{23}$, $C_{13}H_{27}$, $C_{15}H_{31}$

Fig. 1 Synthesis route to glycine and sarcosinebased surfactants

2.2.1. Synthesis of fatty acyl chloride

To take the preparation of dodecanoyl chloride as an example, as shown in fig 1. Dodecanoic acid (100 g, 0.5 mol) was placed in a four-necked flask fitted with a dropping funnel, reflux condenser, mechanical stirrer and thermometer. Hexane was used as a reaction medium in order to ensure completeness of the reaction. 300 ml of hexane was added and the mixture was initially heated upto 45oC till the fatty acid was dissolved. To this, phosphorus trichloride (22.88 g, 0.167 mol) was added dropwise at 50oC. The addition was done over a period of 60 minutes. After the addition was complete, the reaction mixture was heated upto 65-68oC and allowed to reflux for 2.5 hours. After of the reflux period is over, the reaction mixture is stirred at 55oC for another hour. There is generation of yellow solids in the reaction flask. The yellow solids were attributed to the development of phosphorus compounds as the by-product which precipitated out in presence of hexane. The reaction of mixture is allowed to cool and then transferred to a separating funnel. Extreme care must be taken to avoid any yellow solids into the funnel. The upper phase contains the fatty acyl chloride. Hexane and excess phosphorus trichloride is then evaporated off in a rotary evaporator in vacuum, leaving behind a colourless/slight yellowish fatty acyl chloride liquid product. The completion of the reaction was monitored by IR spectroscopy.

2.2.2. Synthesis of Glycinates-12C to 16C and Sarcosinates -12C to 16C

Glycine-12C is taken as an example. Glycine (4.5 g, 0.06 mol) was dissolved in a mixture of acetone and water (2:1). Then 10 % (wt %) NaOH aqueous solution was added dropwise into the mixture until the pH was 9-10. Dodecanoyl chloride (8.74 g, 0.04 mol) was added dropwise with vigorous stirring at 0-5oC. During the reaction process, the pH was kept within a range of 9 to 10 with 10 % (wt %) aqueous solution of NaOH. Then, the mixture was stirred at 0–5oC for another 2 h and allowed to stand at room temperature overnight. Concentrated hydrochloric acid was added to the reaction mixture till the pH was adjusted to 1-2. A white solid precipitated out with the gradual addition of acid. The white solid was vacuum filtered and was with distilled water until the pH of the filtrate was 7. It was further washed with petroleum ether three times. The desired product was recrystallized from ethanol [16]. N-acyl sarcosine were also prepared following a similar protocol except that after neutralization, the surfactant was extracted with ethyl acetate. The N-acyl amino acid products are neutralized by the requisite amount of sodium hydroxide to form different surfactant concentration solutions.

2.3. Titrimetric Analysis of Product

2.3.1. Analysis of raw materials

Acid value of the product/raw material sample was performed as per A.O.C.S official method Te la-64.

2.3.2. Analysis of Fatty acyl chloride

The purity of the fatty acyl chloride formed by the reaction of fatty acid with phosphorus trichloride was ascertained

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following the procedure as described by Chinnick et al. [17]

2.3.3. Analysis of Product

Analysis of the product was done using the official AOCS method using acid value analysis.

2.4 Spectral Studies: FTIR

IR spectra were recorded on Shimadzu (Model No: IR Affinity-1S) FT-IR spectrophotometer. A regular scanning range of 4000– 400 cm-1 was used for 45 repeated scans. All the spectra were recorded with transmission mode.

2.5 Tensiometric measurements

The equilibrium surface tension of surfactant solutions at an air-liquid interface were performed by Krüsstensiometer Model K100 MK2 as evidenced by standard Wilhelmy platinum plate. The measurement error was within ± 0.2 mNm-1. Due to the adsorption of the surfactant on the surface of the plate, there was a fluctuation in reading measurements. To avoid this, the surface was cleaned properly after each measurement. The platinum plate surface was washed first with pure water, acetone, and brought to red hot conditions under the flame to remove the adsorbed surfactants completely. The temperature was maintained throughout the measurements within 30 ± 5 °C by a digital thermostated bath. The cmc values were determined as a function of surfactant concentration by noting distinct breaks.

2.6 Foamability

20 mL of 1% (wt %) surfactant aqueous solution was added into a 100 mL stopper cylinder. Then the stopper cylinder was given 50 strokes. The volume of foam was recorded at 0 min, 5 min, 10 min, and 15 min. Foaminess of other N-acyl amino acid sodium was determined in a similar way.

2.7. Wetting property

Wetting time of surfactant solution at various concentrations was determined using the canvas disc method. 1 % (wt %) surfactant solution is prepared and a canvas disc is placed on the solution's surface. The time recorded from the moment the disc was put into the solution until the moment it started sinking down is the wetting time.

2.8 Emulsion stability

Gly-12C is taken as an example. 20 mL of 1% (wt %) Na-gly-12C aqueous solution, and 20 mL oil phase (dodecane or liquid paraffin) were added into a 100 mL baker. The mixed liquid was stirred by Remi RQG-121/D (equipped with axial turbine) at a speed of 800 rotations/min for 10 min and the emulsion was formed. The emulsion was poured into a measuring cylinder, and the time was recorded when 10 mL lower layer was separated from the emulsion. Emulsibility of other N-acyl amino acid sodium was determined in a similar way.

3. Results& Discussions

3.1 Synthesis

The synthesis of N-acyl amino acid sodium surfactant includes three steps which are the synthesis of long- chain carbonyl chlorides, N-acyl amino acids and sodium salt solution of N-acyl amino acid. Most of the papers [18] [19] [20] discuss the synthesis of fatty acyl chloride using oxalyl chloride, thionyl chloride or phosphorous trichloride in absence of solvent, which can significantly hamper the reaction yield [20]. The reaction can generate a number of phosphorus compounds as undesirable side products. The paper discusses the various side products that can be generated in different temperature, reaction time and reactant ratio conditions. The optimal reaction conditions lie in the temperature of 25 – 70oC. The conversion of fatty acid to fatty acid halide has been carried out in benzene and carbon tetrachlorid. However, because of the carcinogenic nature associated with the use of benzene and carbon tetrachloride as solvent, a potential solvent was sought as a replacement. It was found trial and error that conducting the reaction in hexane helped in the efficient completion of the reaction. The reaction occurs as depicted below

$$3RCOOH + PCI_3 \longrightarrow 3RCOCl + H_3P0_3$$

Using hexane as a reaction solvent was found beneficial. The phosphorus compounds formed as side products separated out as yellow precipitate due to their polar nature. This drastically reduced the separation time of fatty acyl chloride and phosphoric acid. The hexane layer containing fatty acyl chloride was subjected to vacuum evaporation. Hexane subsequently recovered could be used in new batches. The course of the reaction was optimized on the course of the experimentation phase. Temperature controlling is of paramount importance. Changes in temperature can lead to a yellowish final fatty acyl chloride product. Yellowish colour indicated the presence of phosphorus compounds. There are many intermediate phosphorus compounds that are formed along the course of reaction as exemplified by Galbraith. Some of these intermediate phosphorus compounds may have a non-polar character and therefore, very much soluble in hexane phase giving a yellowish fatty acyl chloride. According to specifications, fatty acyl chloride must be a colorless liquid. Such a product could be achieved by following the reaction steps as shown in Table.1

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TABLE 1. REACTION COURSE FOR SYNTHESIS OF FATTY ACYL CHLORIDE

Reaction Phase	Description	Time Duration	Reaction Temperature
I	Dropping phase consisting of addition of phosphorus trichloridedropwise	1 hour	40 – 45oC
II	Reaction carried out at the reflux temperature of hexane	2.5 hour	67– 68oC
III	Temperature controlled within 55oC to limit permanent colour formation	1.5 hour	50 – 55oC

The hydrophobic chain is introduced by acyl chloride. However, the instability of acyl chloride in an aqueous solution is deadly during the preparation process of long chain acyl-amino acid. For acyl-glycine or acyl-sarcosine, the pH must be exactly controlled at 8.5–9.5 with sodium hydroxide, and this pH range is helpful for the nucleophilicity of the amino group and makes it possible to reduce the hydrolysis reaction of acyl chloride as much as possible. Low temperature (~5-10^{o}C) reaction can also avoid the hydrolysis of carbonyl chlorides.

Desired Reaction:

$$RCOCl + H_2NCH_2COOH \longrightarrow RCON HCH_2COOH + HCl$$

Undesired Reaction:

$$RCOCl + H_2O \longrightarrow RCOOH + HCl$$

3.2. Analysis of the raw materials

The fatty acids were analyzed for its purity using acid value titration method.

TABLE 2. PURITY OF FATTY ACIDS

Fatty acid	Purity
Lauric acid	99.03 %
Myristic acid	98.35 %
Palmitic acid	98.61 %

3.2 Analysis of fatty acyl chloride

The purity of fatty acyl chloride was determined by reaction with butanol. Fatty acyl chloride can esterify butanol in absence of catalyst. This reactant is heated on a water bath for 30 minutes. Fatty acid does not esterify butanol in absence of catalyst. Hence, after esterification by fatty acyl chloride, acid value can be used to estimate free fatty acids left unreacted. Hydrochloric acid is neglected, as there was a step of rotary evaporation and fumes of HCl would be eliminated in this step. Fatty acid anhydride is usually reactive and not stable at water bath temperature. As a result, the intermediate fatty acyl chloride was assumed to be free of hydrochloric acid and fatty acid anhydride. The purity of fatty acyl chloride decreased with increasing chain length

TABLE 3. PURITY OF FATTY ACYL CHLORIDE

Fatty acyl chloride	Purity
Lauroyl chloride	86.16 %
Myristoyl chloride	79.23 %
Palmitoyl chloride	77.98 %

3.3. Analysis of the product

Acid value can be used as a method to analyze the product purity. This method assumes that negligible amount of unreacted amino acids are present in the product mixture. After precipitating the product out, it is washed with three times with water that removes free amino acid and subsequently washed with hexane/petroleum ether which removes residual fatty acids. The method estimates the purity of the product using additive principle of acid value. The following calculation is based on lauroyl glycine. Similar methods can be applied to other products.

Acid value of Lauroyl glycine (theoretical) (AV1) = 218.28 mg KOH / g sample
Acid value of Lauric acid (theoretical) (AV2) = 280.5 mg KOH / g sample
AV * 100 = X1 x (AV1) + X2 x (AV2)
Where, X1 = Percentage of Lauroyl glycine
X2 = Percentage of Lauric acid = 100 – X2
AV = Acid value calculated experimentally

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TABLE 4. PURITY OF N-ACYL AMINO ACID

Crude Product	% N-acyl amino acid	% Fatty acid
N-lauroyl glycine	95.31	4.69
N-myristoyl glycine	94.72	5.28
N-palmitoyl glycine	92.11	7.89
N-lauroylsarcosine	93.20	6.80
N-myristoylsarcosine	92.12	7.88
N-palmitoylsarcosine	92.95	7.05

Galaxy Surfactants Inc. is one of the biggest suppliers of N-acyl glycine and N-acyl sarcosine. According to specifications provided by Galaxy surfactants, the chloride content should be low (> 1 %). The chloride content was computed to less than 1 % for all the surfactants.

Yield of the reaction

Schotten-Baumann reaction is used for the acylation across the amino group of the amino acids. The reaction is completed at low temperature and gives quantitative yield results in the range of ~69 – 89 %. The product was purified by recrystallization with ethanol. The yield after recrystallization lied in the range of ~75 - 84 % of the crude product.

TABLE 5. YIELD OF N-ACYLATION REACTION

Reactants	Yield of Crude Product
Glycine + Lauroyl chloride	81.17 %
Glycine + Myristoyl chloride	68.90 %
Glycine + Palmitoyl chloride	79.12 %
Sarcosine + Lauroyl chloride	89.01 %
Sarcosine + Myristoyl chloride	88.02 %
Sarcosine + Palmitoyl chloride	83.87 %

3.4. Fourier Transform Infrared (FT-IR) Analysis

The reaction of fatty acid with phosphorus trichloride leads to some amount of fatty acid remaining unreacted. As the final crude fatty acyl chloride was not distilled, these free fatty acids continue to remain in the crude product. There is no feasible method to analyze the progress of the intermediate reaction. FT-IR was used as a guiding indicator that the reaction was progressing from fatty acid to fatty acyl chloride. The difference between fatty acid and fatty acyl chloride is that the carbonyl group is attached to a hydroxyl group in fatty acids and in case of fatty acyl chloride; the carbonyl group is attached to a chlorine atom. This result in shift of the peaks of carbonyl bond stretching from 1700 cm-1 (fatty acid) to 1800 cm-1 (fatty acyl chloride) and the absence of the broad O–H band of the fatty acid in the region of 3500–2500 cm-1 [22]

TABLE 6. FT-IR ANALYSIS OF INTERMEDIATES

Material	Absorption frequency (observed) (cm-1)	Absorption frequency (theoretical) (cm-1)	Type of vibrations
Fatty acyl chloride	1797.66	1800	C=0 stretching of fatty acyl chloride
Fatty acid	1697.36	1725 - 1700	C=0 stretching of fatty acid

The reaction was monitored through FT-IR of the sample taken out at 2.5 hours. This has depicted in the FT-IR spectra in fig 2the diminishing peak at 1705.07 cm-1 indicates reaction is progressing towards the formation of fatty acyl chloride.

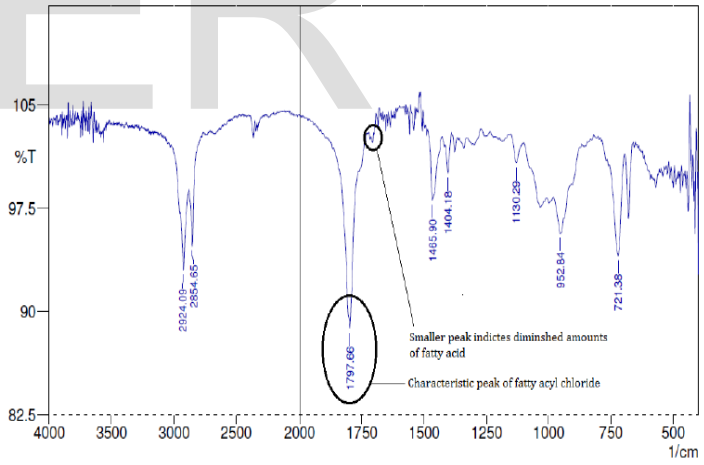

Fig 2 FT-IR spectra of Lauroyl chloride

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3.4.1FT-IR spectral analysis of N-acyl amino acid

TABLE 7. FT-IR ANALYSIS OF FINAL PRODUCTS

Material	Absorption frequency (observed) (cm^{-1})	Absorption frequency (theoretical) (cm^{-1})	Type of vibrations
Ester Group	1720.15	1720-1750	Stretching
C-N Group	1462	1410-1470	Bending
C-H group	2922.10-2855-72	2930-2850	Stretching

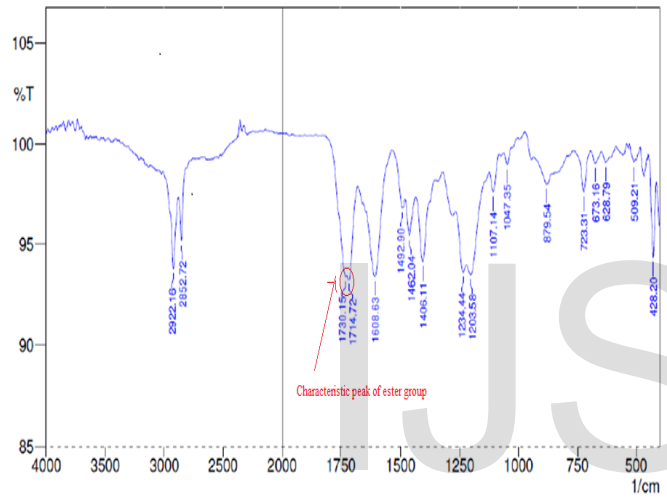

Fig 3. FT-IR spectra of N-lauroyl glycine

Formation of an N-acyl glycine surfactant was confirmed by the presence of the peaks at 1462, 1620 and 1720 cm-1 in Fig 4, which are attributed to the C–N, C=O and COO- groups, respectively [22]. Besides, it shows an intense C-H stretch at 2922 cm-1 and 2852 cm-1 that corresponds to the long fatty acid chain.

3.5. Tensiometric measurements

The cmc and γcmc are important parameters for surfactants. The surface tension of the two series surfactants at different concentrations was measured by Wilhelmy plate technique at 30 + 5 oC. The curve of surface tension versus concentration was drawn to obtain cmc and γcmc, which were shown in Table 6.

TABLE 8. CMC AND ΓCMC OF AMINO ACID BASED SURFACTANTS

Surfactants	Critical Micelle Concentration	Surface Tension @ cmc
	CMC (mmol/l)	γ_{cmc}(mN/m)
Sodium LauroylGlycinate	18.90	33.136
Sodium MyristoylGlycinate	4.51	36.112
Sodium PalmitoylGlycinate	0.53	26.896
Sodium LauroylSarcosinate	7.18	29.536
Sodium MyristoylSarcosinate	0.47	22.610
Sodium PalmitoylSarcosinate	5.43	22.239

There are general rules of amphipathic molecule homologues: with the lengthening of hydrophobic chain, the efficiency of reducing surface tension increases and cmc decreases. In glycinate series of surfactants, the cmc decreases with increasing chain length, whereas, for sarcosinate surfactants, cmc reaches minimal value for C14 and then there is again a subsequent increase in cmc. The cmc and γcmc values reported for glycinate surfactants differ in different papers [23][24]. The reason may be that these amino acid based surfactants are pH sensitive. The surface activity can change drastically with changes in pH as shown in the product brochure by Chattem chemicals Inc. for their Hamposyl® range of surfactants. The surface tension of sodium cocoylsarcosinate varies from 22 mN/m to 30 mN/m during the pH change from 8 to 9.

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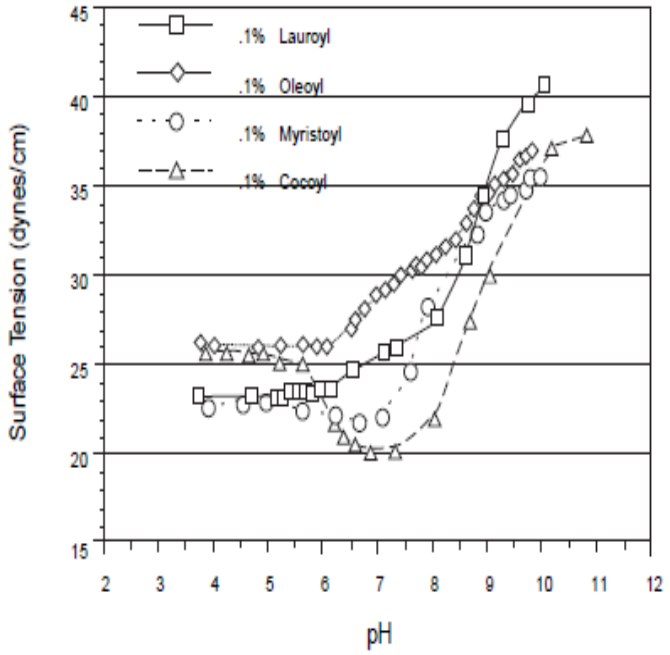


Fig 4. Surface activity of N-acyl sarcosinate with respect to pH changes [24]

In the procedure of tensiometric measurements, a suitable amount of water is taken and its surface tension is initially analyzed by the Wilhelmy plate method. Then an entered amount of surfactant is added to the water and again a measurement is computed. This procedure goes on repeating till the number of steps is completed as desired by the operator. N-acyl amino acid is neutralized by requisite amount of sodium hydroxide and the pH of this solution is usually ~9. However, when a small amount of this surfactant solution is added to the water in the first step, the pH of the mixture changes and it is not equivalent to 9. As a result, at different additions pH keeps increasing. The information from Chattem Inc. clearly demonstrates the drastic effects of pH on the surface tension of the surfactant solution. This may be one of the reasons why for tensiometric measurements of some surfactant solution went haywire at different additions

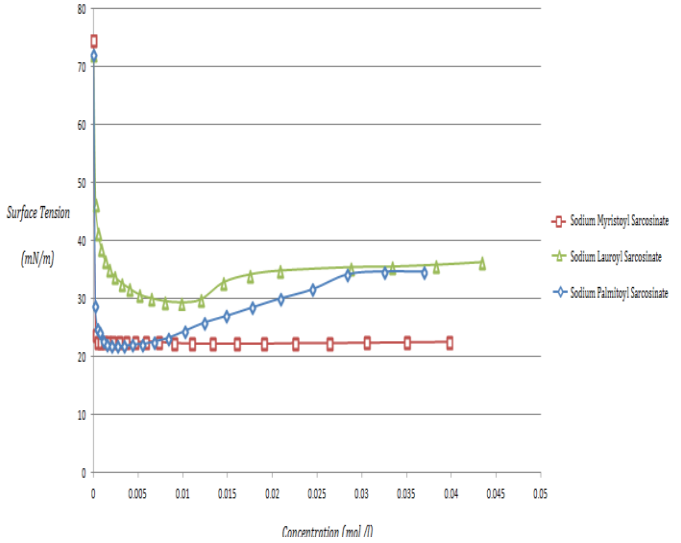
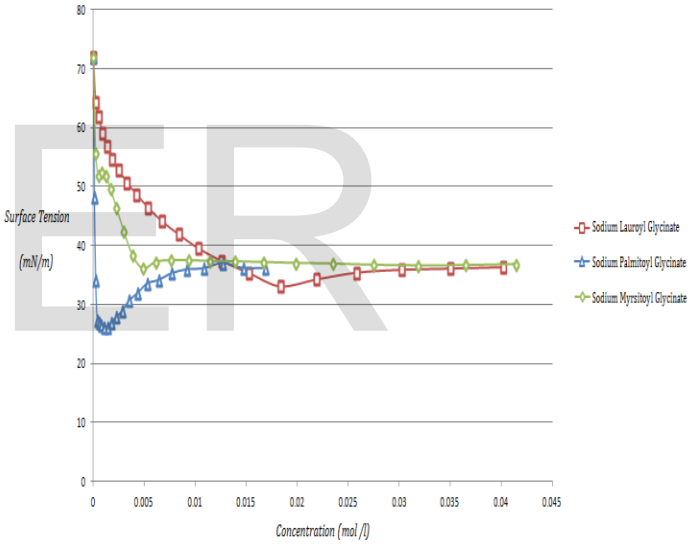


Fig .5. Surface tension versus concentration of surfactant at 30 + 5oC



FFig .6 Surface tension versus concentration of surfactant at 30 + 5oC

3.6. Foamability

The study of surfactants' foamability mainly aims at washing process. Foam can strongly adsorb dirt, thus effectively preventing dirt returning to washing surface. Though there are no studies showing direct relationship between foam and washing ability, the foam is indispensable in the washing process. It is one of the indexes for application of surfactants. For glycinate series of surfactants, foamability got better as the hydrophobic chain length increased. Sodium lauroylglycinate exhibited good volume of initial foam. But, the foam stability is not appreciable, as foam settles down quickly within time duration of 1-2 minutes. The foam stability also shows to increase with the chain length. Sarcosinate based surfactants are excellent foaming agent. Copious and stable foam is a characteristic of sarcosine based surfactants. It is noted that a small difference of methyl group yields different surfactant

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properties.

TABLE 9.FOAMABILITY OF GLYCINE AND SARCOSINE SURFACTANTS

Surfactants	Concentration (wt %)	Volume of foam (ml) after:			
		0 min	5 min	10 min	15 min
Sodium Lauroyl Glycinate	0.5	72	0	0	0
	1.0	40	0	0	0
Sodium MyristoylGlycinate	0.5	80	35	35	25
	1.0	87	55	30	25
Sodium PalmitoylGlycinate	0.5	68	68	68	68
	1.0	71	71	71	71
Sodium LauroylSarcosinate	0.5	60	60	60	60
	1.0	65	60	60	60
Sodium MyristoylSarcosinate	0.5	103	103	103	103
	1.0	112	110	110	110
Sodium PalmitoylSarcosinate	0.5	58	58	58	58
	1.0	65	65	65	65

3.7. Wetting Time

The wetting power of individual series of surfactants was performed by the Canvas disc method. Lower the wetting time, better is the surfactant efficiency to completely wet a surface. Sodium myristoylsarcosinate exhibits very low wetting time of 11 seconds. For the glycine surfactant series, the wetting time increases as the chain length increases.

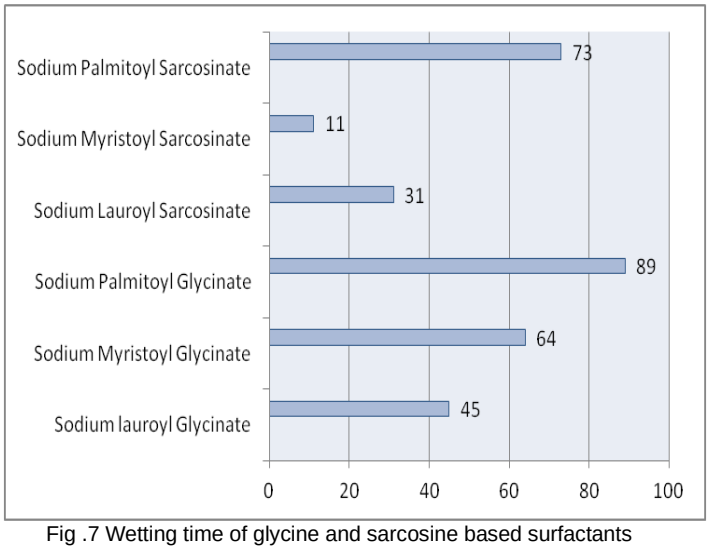

Fig .7 Wetting time of glycine and sarcosine based surfactants

3.8. Emulsion Stability

It is defined as emulsification that one liquid is dispersed into another liquid in the form of liquid droplets. After emulsification, large interface between two liquids (eg. water and oil) was formed, resulting in the increase of free energy. Therefore emulsion is a thermodynamically unstable system. The formation of stable emulsion needs surfactants. The interface tension between water and oil phase is reduced, and the emulsion will more stable. Therefore, it is important to study the emulsion stability of surfactants.

In this work, liquid paraffin was chosen as oil phase. The surfactants concentrations were 1% (wt %). The volume of aqueous solution and oil phase were 20 ml respectively. The time for the separation of 10 ml from the lower aqueous layer was noted.

TABLE 10.EMUSIBILITY OF GLYCINE AND SARCOSINE BASED SURFACTANTS

Surfactants	Concentration (wt %)	Liquid Paraffin
Sodium LauroylGlycinate	1.0	1 min 21 sec
Sodium MyristoylGlycinate	1.0	3 min 19 sec
Sodium PalmitoylGlycinate	1.0	2 min 51 sec
Sodium LauroylSarcosinate	1.0	4 min 4sec
Sodium MyristoylSarcosinate	1.0	2 min 2 sec
Sodium PalmitoylSarcosinate	1.0	1 min 35 sec

4. CONCLUSION

Two series of surfactants with different amino acid moiety (glycine, sarcosine) were synthesized. The reaction processes were simple and the conditions were mild. Their surface activity and foamability were studied. The cmc and γcmc decreased with increasing chain length. Glycine based surfactants exhibited strong initial foam. However, the foam volume decreased with time (unstable foam). Sarcosine series of surfactants exhibited excellent foaming capacity and foam stability. The foam was stable over a period of half an hour. This demonstrates its ability to be used in personal care formulations such as shampoos, which require copious amount of foam. Both the series of surfactants do not perform well with respect to emulsion stability. Being derived from natural amino acids, these types of products also become a source of marketability to the consumers. Amino acid based surfactants require renewable raw materials such as fatty acid (derived from vegetable oil) and amino acid (derived from protein). As a result, amino acid surfactants are mild in nature and have the ability to work in synergism with other

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surfactants as well.

5. Scope for future works

Every chemical material or process is scrutinized for safety and compatibility to minimize impact inorder to minimize the increasing concern for the effect of industrialization on human life and the environment. Surfactants are functional chemicals in principle; so many synthetic surfactants are developed to improve their cost effectiveness.

The cost of amino acids and proteins and complicated manufacturing processes were the prohibiting factors for the broad utilization of this class of surfactants. However, large-scale production and market consumption of certain amino acids, such as glutamic acid and glycine, have helped to alleviate this limiting factor. The combination of synthetic and biological processes will be another prospective driving force to help reduce the production cost of amino acid and protein-based surfactants.

One of the major difficulties to the commercialization of amino acid based surfactants is the high cost associated with the product manufacturing. The large cost of raw material (amino acid and fatty acid halide) is a limiting factor. Pure amino acid can be replaced with amino acids obtained from protein hydrolysates. This will also lead to efficient by-product utilization of the oil meal. The same reaction of acylation can be carried out using fatty acid methyl esters. However, reaction involving fatty acid methyl esters demand a slightly high pressure and higher temperature. Therefore, a thorough economics of the process needs to be carried out before upscaling. Fatty acid methyl esters would also potentially replace the use of harmful chlorinating agents such as phosphorus trichloride or thionyl chloride.

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