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Synthesis, characterization and surface properties of bis-sulfonamide, sulfonamide-imine and sultam based nonionic surfactants

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Abstract

In an effort to develop new classes of nonionic surfactants, the surface tension properties and phase behavior of a novel series of bis-sulfonamide, sulfonamide-imine and sultam-surfactants have been investigated. These surfactants possess SO2NH moieties that display comparative cytotoxicities. In this paper the presence of the –SO2NH functional moiety and the partition coefficient (oil/water) measurement demonstrate good biodegradability and lipophilicity. On the other hand, by means of the located thermodynamic point, the solubilization can be considered as a normal partitioning of the drug between two phases, micelle and aqueous. A direct relationship has been found between the efficiency and solubility.

Keywords: Hinder chemistry, Conjugative stabilization, Schrödinger equation/Darwinism, Isoelectronium, Nonionic surface-active agents.

INTRODUCTION

In French, German or Spanish languages the word "surfactant" does not exist, and the actual term used to describe these substances is based on their properties to lower the surface or interface tension, e.g. tensioactif (French), tenside (German), tensioactivo (Spanish). This would imply that surface activity is strictly equivalent to tension lowering, which is not absolutely general, although it is true in many cases.

Nonionic surface-active agents have different partition coefficients. They can be divided into three categories: oil soluble, water dispersible, and water-soluble. They are generally insoluble in water and are used as water-in-oil emulsifiers and as wetting agents [1].

Reduction of surface and interfacial tension

When surfactants are dissolved in water they orientate at the surface so that the hydrophobic regions are removed from the aqueous environment. The reason for the reduction in the surface tension when surfactant molecules adsorb at the water surface is that the surfactant molecules replace some of the water molecules in the surface and the forces of attraction between surfactant and water molecules are less than those between two water molecules, hence the contraction force is reduced.

Surfactants will also adsorb at the interface between two immiscible liquids such as oil and water and will orientate themselves with their hydrophilic group in the water and their hydrophobic group in the oil [2].

The interfacial tension at this interface, which arises because of a similar imbalance of attractive forces as at the water surface, will be reduced by this adsorption.

The surface activity of a particular surfactant depends on the balance between its hydrophilic and hydrophobic properties. For a homologous series of surfactants: An increase in the length of the hydrocarbon chain (hydrophobic) increases the surface activity [3].

Drug absorption is influenced by many biological and physicochemical factors. The two most important physicochemical factors that affect both the extent and the rate of absorption are lipophilicity and

solubility [4]. Similarly, Daniel *et al.* have shown that the absorption rates of a series of cationic drugs in rat small intestine correlated well with their lipophilicity [5]. The membrane of the gastrointestinal epithelial cells is composed of tightly packed phospholipids interspersed with proteins. Thus, the transcellular passage of drugs depends on their permeability characteristics to penetrate the lipid bilayer of the epithelial cell membrane, which is in turn dependent on the lipophilicity of the drugs. The effect of lipophilicity on the absorption rates is best exemplified by the classical study of barbiturates conducted by Zhong [6]. The effects of lipophilicity on membrane permeability and first-pass metabolism appear to have opposing effects on the bioavailability. Thus, it is important to balance the effects of lipophilicity on membrane permeability and first-pass metabolism to improve bioavailability [7].

In studies with structure related sulfonamides, Yuan *et al.* have shown that there was a strong correlation between plasma protein binding of the drugs and their lipophilicity [8]. Monish *et al.* found that the volume of distribution increased with increasing lipophilicity when administering a series of sulfonyl drugs to selected tumor cell lines [9]. The influence of lipophilicity on the metabolic clearance of drugs is attributed mainly to the increased affinity of drugs for the enzymes [10].

Also, it should be pointed out that there are many factors, in addition to lipophilicity, that can influence first-pass metabolism. Solubility is an important determinant in drug absorption; a drug must be reasonably soluble in the aqueous environment to be absorbed properly, i.e. highly lipophilic and poorly soluble, resulting in poor bioavailability. Consequently, the drug Solubility will depend on its polarity: nonpolar molecules will be solubilized in the micellar core, and substances with intermediate polarity will be distributed. Micellar systems of drugs can increase their bioavailability, they can stay in the body (blood) long enough to provide gradual accumulation in the required area, and their sizes permit them to accumulate in areas with leaky vasculature [12].

MATERIALS AND METHODS

Materials

Six novel functionalized bis-sulfonamide, sulfonamide-imine and sultam surfactants were synthesized according to published methods [3]. DMSO-d_6 was obtained from Cambridge Isotope Laboratories. N-acetyl sulfanilyl chloride, 1,4-amino-9,10-anthraquinone, dodecyl amine, 3-methyl-1H-pyrazol-5(4H)-one, 3-cyano-1H-pyrazol-5(4H)-one, 1H-Pyrazol-5(4H)-one were obtained from El Nasr Chemical CO.

Instrumentation

Microelemental analysis (C, H, S, X) was performed via Varian Elemental instrument and the samples were drawn in satisfactory agreement with the calculated values. ^{1}H-NMR spectra were recorded on Varian Gemini (200 MHz) instrument and the samples were run in deuterated dimethyl sulfoxide, DMSO-d_6, corresponds to tetramethyl silane as an internal standard. FT-IR spectra were designated as Perkin-Elmer spectrum in one spectrophotometer 4,000 - 400 cm^{-1} of KBr discs.

The surface tension (γ) of the surfactant solutions (of concentration range of 0.1 to 0.0001 m/L) was measured with a Krüss K-6 tensiometer (Hamburg, Germany) based on the Du- Nouy platinum ring method and was calibrated using deionized water at 25° C. The platinum ring was cleaned before each measurement by flaming Bunsen (burner) to remove any residual deposit.

The surface tension measurements were taken after 90 min; of pouring the surfactant solution in the measuring container to ensure the equilibrium occur. The readings were taken in triplicate for each solution to check repeatability and the surface tension values were measured within an error less than or equal to ±1 mN/m. Interfacial tension measurements (σ) were carried out for 0.1 g/l surfactant solutions in paraffin oil at 25° C [12].

Methodology of P_{ow}

The partition coefficient is simply the ratio of the equilibrium concentrations between the two immiscible phases in contact, i.e. "The ratio of a chemical's concentration in the octanol phase to its concentration in the aqueous phase of a two-phase octanol/water system" [13].

P_{ow} = Concentration in octanol phase/Concentration in aqueous phase

The volume ratio of octanol and water mixture is adjusted according to the expected value of P_{ow} (< 3).

1) The concentration of the solute in the system was less than 0.001 mol/L in any single phase.

2) Very pure octanol and water were used.

3) The system, usually in a separator funnel was shaken gently until equilibrium is achieved. The system is then centrifuged to separate the two phases and break any emulsions.

4) The two phases are then analyzed by an appropriate technique, UV-Vis, to determine solute concentrations. Both phases were analyzed to achieve mass balance.

5) To evaluate the concentration of solute in two phases, different moles of solute were used in the range of 0.01-0.03 mM, holding constant the value of the wavelength (λ of that sample), the corresponding absorbance were investigated.

6) The partition coefficient, P_{ow}, of solute in two phases was described as

P_{ow} = Concentration of solute in octanol phase / Concentration of solute in aqueous phase.

Product identification

The elemental analysis showed the purity of the synthesized compounds (99.7%). The IR and ^{1}H-NMR spectra showed the chemical structures of the synthesized surfactants which are identical as represented in the following:

Compound I (MW= 600): yield 87%. IR: 3264, 2889-2200, 1720, 1154 cm^{-1} corresponded to (NH, CH, C=O, S=O stretching). ^{1}H-NMR: d = 8 ppm (s, 1H, α-C(=O)NH), 4 ppm (s, 1H, α-S(=O)NH), 2.02 ppm (s, 3H, $-CH_3$), 6.5-8 ppm (m, $4H_{AROMATIC}$, 4 rings). Cal. (Found %): C, 60 (59.9 %), H, 4 (3.9 %), O, 16 (15.9 %), N, 9 (8.9 %), S, 11 (10.9 %).

Compound II (MW= 575): yield 78%. IR: 3300, 2886-2400, 1715, 1600, 1100 cm^{-1} (NH, CH, C=O, C=N, S=O stretching groups); ^{1}H-NMR: d = 0.9 ppm (s, 3H, CH_3), 4 ppm (s, 1H, α-S(=O)NH), 1.3 ppm (m, 2H, CH_2), 7.3 ppm (m, $4H_{AROMATIC}$, 3 rings), 4 ppm (s, $1H_{AMIDE}$). Cal. (Found %): C, 69 (68.9 %), H, 7 (6.9 %), O, 11 (10.9 %), N, 7 (6.9 %), S, 5.6 (5.5 %).

Compound III (MW= 549): yield 72%. IR: 4320, 2200, 1600, 1100 cm^{-1} corresponded to (NH, CH, C=N, S=O str.); ^{1}H-NMR: d = 1.3 ppm (s, 2H, CH_2), 0.9 ppm (s, 3H, CH_3), 4 ppm (s, $1H_{AMIDE}$), 2 ppm (s, 1H, α-S(=O)NH), 6.5-8 ppm (m, $4H_{AROMATIC}$). Cal. (Found %): C, 70 (69.9 %), H, 11 (10.9 %), N, 8 (7.9 %), O, 6 (5.9 %), S, 6 (5.9 %).

Compound IV (MW= 295): yield 82%. IR: 3977, 3100, 1725, 1600, 1100 cm^{-1} corresponded to (NH, CH, C=O, C=N, S=O str.); ^{1}H-NMR: d = 0.9 ppm (s, 3H, $_{ANCORED}$-CH_3), 8 ppm (s, 1H, α-C(=O)NH), 2.02 ppm (s, 3H, CH_3), 2.2 ppm (s, 2H, $_{ANCORED}$-CH_2), 7.9 ppm (m, $4H_{AROMATIC}$). Cal. (Found %): C, 48.8 (48.7 %), H, 4.4 (4.3 %), N, 14.2 (14.1 %), O, 21.7 (21.6 %), S, 10.9 (10.8 %).

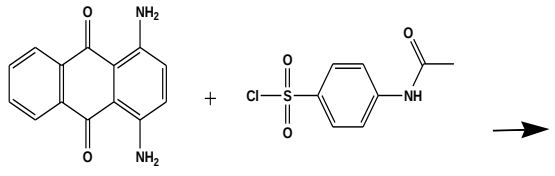
Compound V (MW= 306): yield 75%. IR: 2950, 2842, 1735, 1600, 1100 cm^{-1} corresponded to (CH_2, C=O, C=N, S=O str.); ^{1}H-NMR: d = 2.02 ppm (s, 3H, CH_3), 8 ppm (s, 1H, α-C(=O)NH), 2.2 ppm (s, 2H, $_{ANCORED}$-CH_2), 7.9 ppm (m, $4H_{AROMATIC}$). Cal. (Found %): C, 47 (46.9 %), H, 3.3 (3.2 %), N, 18.3 (18.2 %), O, 20.9 (20.8 %), S, 10.5 (10.4 %).

Compound VI (MW= 462): yield 80%; IR: 2950, 2842, 1735, 1600, 1100 cm^{-1} corresponded to (CH_2, CH_3, C=O, C=N, S=O str.); ^{1}H-NMR: d = 2.2 ppm (s, 2H, $_{ANCORED}$-CH_2), 8 ppm (s, 1H, α-C(=O)NH), 7.9 ppm (m, $4H_{AROMATIC,}$ 2 rings), 2.02 ppm (s, 3H, CH_3). Cal. (Found %): C, 49 (48.9 %), H, 4 (3.9 %), N, 12 (11.9 %), O, 20 (19.9 %), S, 14 (13.9 %).

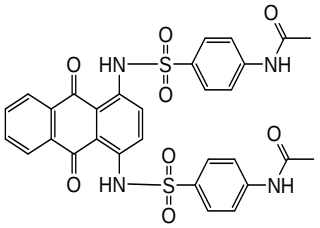
Synthesis

Synthesis of the bis-sulfonamide surfactants (I)

A flat container was charged with (0.02 mol) 4.68 gm of N-acetyl sulfanilyl chloride and 10 ml of methylene chloride under cold condition, and then (0.01 mol) 2.38 gm of 1, 4-amino-9, 10-anthraquinone were added gradually with stirring. After the addition was completed, stirring was continued for 6 h at room temperature, and then washed thoroughly with distilled water and a deposited yellow product (I) was obtained.



1,4-diamino-9,10-anthraquinone N-acetyl sulfanilyl chloride

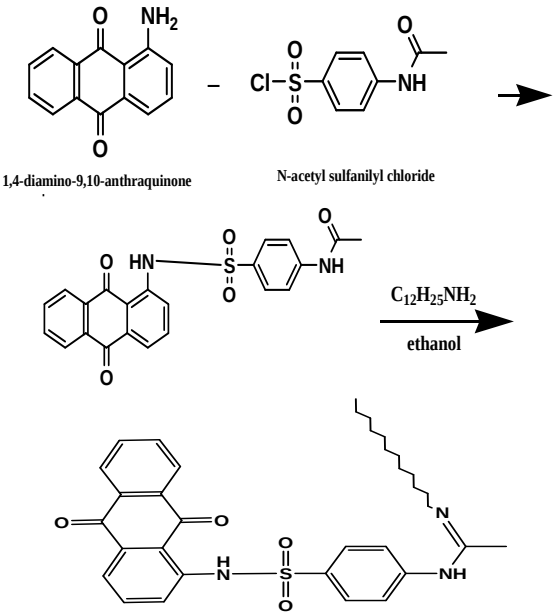


***N,N*-{4-[(4-amino-9,10-dioxo-9,10-dihydroanthracen-1-yl) sulfamoyl]phenyl}diacetamide (I)**

Synthesis of the Ar-sulfonamide-imine surfactants (II)

A solution of 1-amino-9, 10-anthraquinone (0.01 mol, 2.33 gm) and N-acetyl sulfanilyl chloride (0.01 mol, 2.34 gm) in DMF was stirred at room temperature for about 2 h, and then neutralized by a 0.1 N of sodium hydroxide. The mixture was refluxed for 3 h. The resulting product was separated and crystallized by methanol to remove the excess of hydrochloric acid.

A solution of sodium salt of the sulfonamide derivative (0.01 mol) was added to (0.01 mol) solution of dodecyl amine ($C_{12}H_{25}NH_2$) and 2-3 drops of conc. H_2SO_4. The mixture was heated at reflux for 7 h (70° C). The combined organic fractions were concentrated in *vacuo* to yield a pale yellow solid that was recrystallised with methanol to give the title compound.

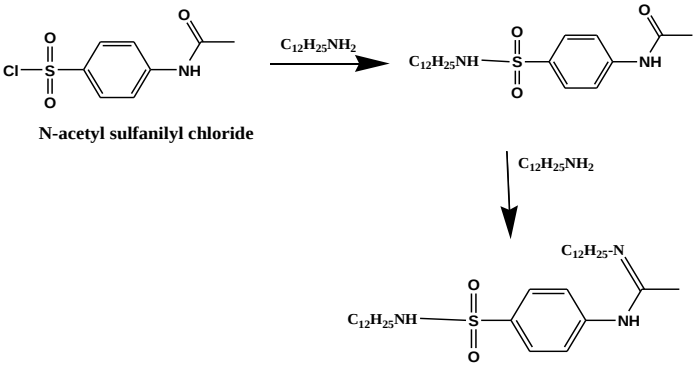


1,4-diamino-9,10-anthraquinone N-acetyl sulfanilyl chloride

***N*-{4-[(9,10-dioxo-9,10-dihydroanthracen-1-yl)sulfamoyl] phenyl}dodecylethanimidamide (II)**

Synthesis of the aliph-sulfonamide-imine surfactants (III)

A mixture of N-acetyl sulfanilyl chloride (0.01 mol, 2.34 gm), dodecyl amine (0.01 mol, 1.85 gm) and Pyridine (0.01 mol) in diethyl ether (50 mL) was heated under reflux for 4h. The solvent was then evaporated under reduced pressure and some cold water (20 mL) was added. The solution was extracted with ethyl alcohol (100 mL) to remove the excess of hydrochloric acid. A solution of sodium salts of sulfonamide derivatives (0.01 mol, 3.83 gm) was added to (0.01 mol) solution of dodecyl amine ($C_{12}H_{25}NH_2$) and 2-3 drops of conc. H_2SO_4 and the mixture was refluxed for 7 h. The combined organic fractions were concentrated in *vacuo* to yield a pale yellow solid that was recrystallised with methanol to give the title compound.



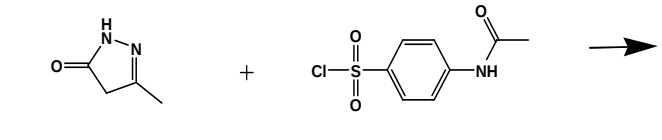
N-acetyl sulfanilyl chloride

N,N-dodecyl-N-[4-(methylsulfamoyl) phenyl]ethanimidamide (III)

Synthesis of the sultam surfactants (IV, V)

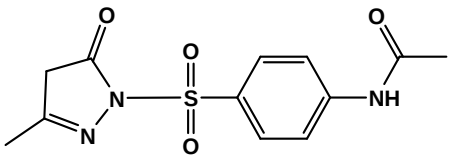
0.01 mol (1 gm) of pyrazoline derivatives were refluxed for 8 h with 0.01 mol (2.34 gm) of N-acetyl sulfanilyl chloride in presence of THF (50 mL) and 2-3 drops of conc. H_2SO_4 were added to the reaction mixture and then allowed to separate overnight. The produced salts

were filtered off, washed twice with acetone and dried at 40° C in *vacuum* oven.

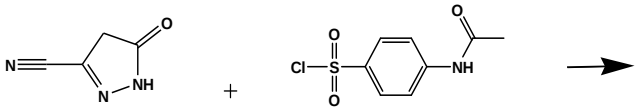


3-Methyl-1H-Pyrazol-5(4H)-One

N-acetyl sulfanilyl chloride

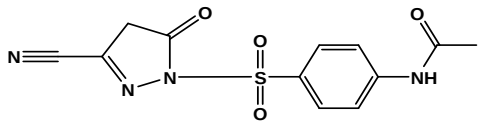


N-{4-[(3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-1-yl)sulfonyl]phenyl}acetamide (IV)



3-Cyano-1H-Pyrazol-5(4H)-One

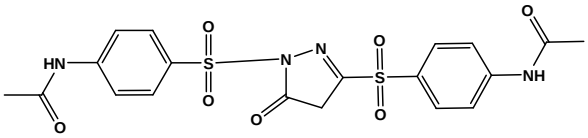
N-acetyl sulfanilyl chloride



N-{4-[(3-methyl-5-oxo-4,5-dihydro-1H-pyrazol-1-yl)sulfonyl]phenyl}acetamide (V)

Synthesis of the sultam surfactants (VI)

0.01 mol of 1H-Pyrazol-5(4H)-one was refluxed with 0.02 mol of N-acetyl sulfanilyl chloride and 2-3 drops of conc. H_2SO_4 were added in presence of triethyl amine (50 mL) for 8 h and allowed to separate overnight. The produced salt was filtered off, washed twice with acetone and dried at 40° C in *vacuum* oven to give compound (VI).



N,N'-[(5-oxo-4,5-dihydro-1H-pyrazole-1,3-diyl)bis(sulfonylbenzene-4,1-diyl)] diacetamide (VI)

RESULTS

The titled compounds were comparable with respect to P_{ow} (octanol/water partition coefficient of the solutions), γ_{cmc} (surface tension of the solutions), σ_{cmc} (interfacial surface tension of the solutions) (Table 1). The treatment was done for each sample at 0.02 mol/L and was comparable prior to each relationship. The higher the lipophilicity, the higher antitumor activity as shown in Table 1 ($P < 3$).

Table 1: Surface properties of the desired compounds

Compound	I	II	III	IV	V	VI
γ mNm^{-1}	54	31	45	23	28	58
σ mNm^{-1}	16.3	14	15	9	14	==
Log P_{ow}	0.55	0.37	0.97	0.77	0.86	1.0

DISCUSSION

Behavior of the bisect dienophile as an electron donor

The behavior of the dienophile as an electron donor is a final point within this research. It should be noted that the Cplex-isoelectronic theory, in its present qualitative form, is like all qualitative theories, prone to some degree of posterior rationalization in some cases [14-18]. This can be seen, for example, in deciding whether a thermal reaction can occur via a concerted electronic mechanism or by a stepwise pathway.

It is not necessarily, according to Dewar; a limitation in juxtaposition by a priori quantum mechanical calculation [19]. Some of these a posteriori rationalizations can be removed when more knowledge about the experimental data, on which the assumptions are deduced from, is discovered. According to, Hanan; all present synthesized methods are wholly empirical and is sufficient by comparing with experimental [3].

This resulted from the fact that it is impractical to affect an exact representation for complex chemical systems, especially chemical reactions, based on the present methods. On this respect the emergence of $NHCOCH_3$ chemistry is highly promising as convergence occurs at least 10 times faster than the present Cplex-isoelectronic (C.I.) participations [20]. Exploring the consistency between the predictions of the Cplex-isoelectronic theory and hinder chemistry is for future study.

As hinder chemistry permits an exact quantitative representation of the chemical bond it can make new predictions for pericyclic reactions and aromatic compounds. The involving multiple bonds (no bridging intermediate possible as H involved) is highly stereospecific and syn, syn is increasingly favored at low temperatures and by weakly donating solvents as well as by increasing the concentration of HX [21].

Furthermore, the nucleophilic, radical addition, SN2 and SN2' reactions (B and B' assumptions) are deduced from 1, 3-pyrazoline substitution reactions and the anomalous effect, in line with the Robinson/Ingold electronic theory.

Application of the heavy atom effect provides experimental evidence for the synperiplanar dynamics of isoelectron pairs, SDSE, mode in pericyclic reactions.

The present quantum chemical theories require the addition of a small correction factor for molecules with two or more electrons namely, SDEP, synperiplanar dynamics of isoelectron pairs; RCEDGD, Rate of continuous electron dynamics greater than delocalisation. Based on the above logic it is feasible that these quantum-based approaches make imprecise predictions for these complex chemical systems. The Cplex-isoelectronic theory, like Darwinism and the Robinson/Ingold theory, is qualitative, making logical and scientific connections between known and unknown systems (within the chemical level), an approach seen in the emerging area of Complexity theory [22]. There is still a place for such qualitative theories for complex systems considering the above discussion in relation to the present quantum chemical methods. In fact Fukui stated that "chemistry inevitably depends on analogy through experience due to the formidable complexity". As this new theory is based on the chemical and not the quantum level it is less prone to making inaccurate predictions as there are less factors involved. Expansion into the hadronic and quantum level is for the future, either in a qualitative or quantitative form, when the theory is fully developed on the chemical level [23].

Since biological membranes are lipoidal in nature, the rate of drug transfer for passively absorbed drugs is directly related to the lipophilicity of the molecule. The lipophilicity of a drug not only affects the membrane permeability, but the metabolic activity as well [23-26]. The greater its metabolic clearance, the greater its first-pass metabolism is. In conclusion, local anesthetics with high lipophilicity are highly toxic and induce mainly necrosis, while those with low pKa more induce apoptosis.

Although it appears that the partition coefficient may be the best predictor of absorption rate, the effect of dissolution rate, pKa, and solubility on absorption must not be neglected. The higher lipophilicity (more toxic) of compound VI affects its absorption, metabolism, its binding and distribution compared with those structure-lipophobicity relationships for I, II, III, IV, V sulphonamide-drugs.

CONCLUSION

The present study reported the octanol/ buffer coefficient (log P value) as the lipophilicity of the drug and the clinical potency correlated well with fatality rates, (LD_{50}), as well as compound VI may facilitate its combination with the tumor cell lipids and interferes with its enzyme balance.

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