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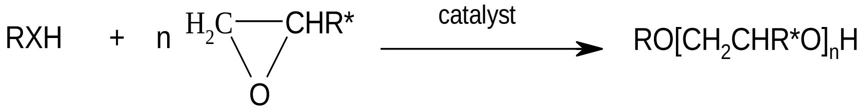
Synthesis of Nonionic Surfactants by Treating Some Petroleum Derivatives

K. A. Hebash , M. H. M. Ahmed and G. F. El-Hady
Chemistry Department, Faculty of Science, Benha University,
Benha, Egypt.

DODECYL benzene sulphonic acid was used in preparation of a series of nonionic surfactants (II - V) a-c. The preparation was completed by the reaction of dodecyl benzene sulphonic acid with thionyl chloride followed by the alkylene diamine series (n = 2, 4, 6, 8) to give Ia-d, respectively. The later products (Ia –d) were reacted with ethylene oxide to produce nonionic surfactants (II - V) a-c. The structures of the prepared nonionic surfactants were confirmed by IR and ^{1}HNMR spectra. The surface properties, biodegradability and biological activities of the prepared compounds were investigated. The obtained data show that these compounds have good surface and biological activities besides, reasonable biodegradability properties.

Nonionic surfactant is one of the most important groups of surfactants with growing industrial interest, it can be synthesized by ethoxylation (addition with ethylene) of hydrophobic organic compounds containing active hydrogen in the presence of traditional catalyst (conventional basic catalysts, *e.g.* NaOH, or acids, *e.g.* BF_3, …etc. (1,2) rather than untraditional catalyst *e.g.*, different clays, calcium compounds, …etc.) (3, 4).



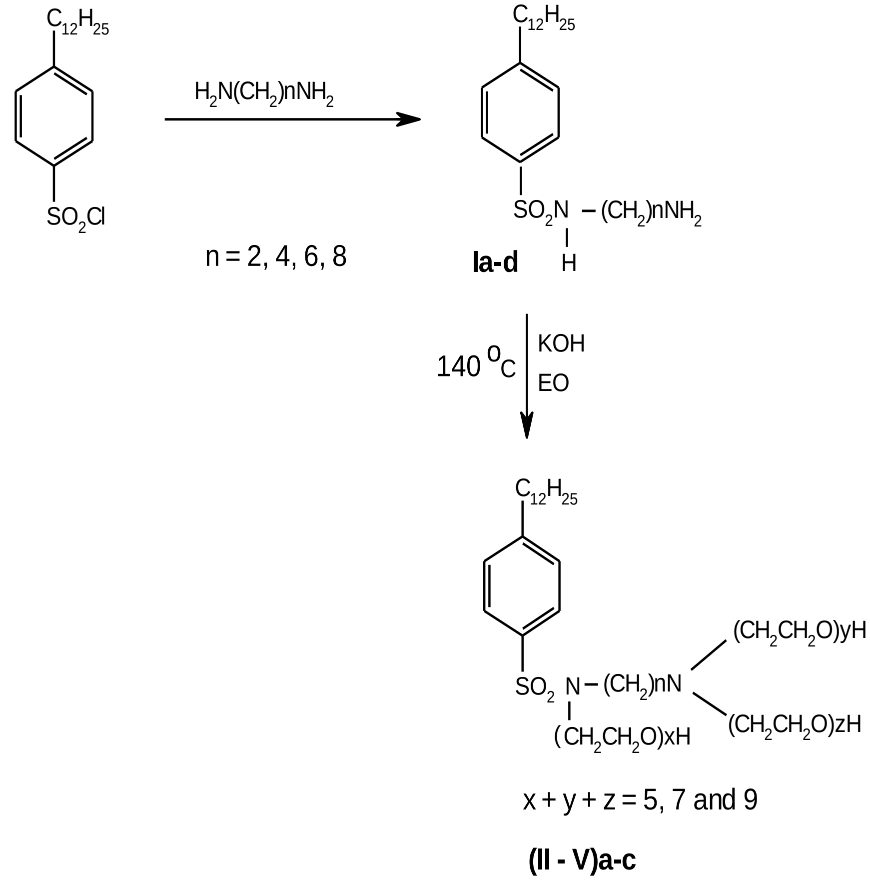
where: R is long chain aliphatic hydrocarbon, alkyl phenyl, aralkyl group
XH is OH, SH, COOH, NH, etc.
R* is H (ethoxylation) or CH_3 (propoxylation);
n is moles of alkylene oxide reacted with one mole of starting material

The product of the ethoxylation is a mixture of oligo(poly)ethylene glycol ethers (or esters). Therefore, the properties of nonionic depend on the ethylene oxide added and the type of starting molecules.

Ethoxylation is a process extensively used by industry to produce a large number of nonionic products. An important class of such products are poly (ethylene glycol)s, that is largely used as final or chemical intermediates, lubricants, industrial surfactants, and components for cosmetics and personal care formulations (5).

The hysrophilic group generally is an oligopolyethylene glycol chain with a free hydroxyl or amino group at the end of molecule. Hydrophobic group is usually a long chain of the fatty alcohol, acid, alkylphenol or aralkyl alcohol (6). Using hydrophobic part like dodecylbenzene suphonyl choloride of commercial and technical grade is the target of this work.

The aim of this work is to prepare nonionic surfactants from some crude oils. Dodecyl benzene sulphonic acid is one of the most popular and cheap crude material used in the synthesis of a large number of surface active agents. So, this compound is used for the synthesis of some surface active agents containing sulphonamide group with expected biologically by active properties. The reactions are shown in Scheme 1.



Scheme 1.

Materials and Methods

Dodecyl benzene sulphonic acid (Technical grade) was supplied from Nasr Comp.; thionyl chloride (Merck); epichlorohydrine (Aldrish); triethanol amine (Technical grade); triethyl amine (Merck); 1,2-ethylene diamine (Merck); 1,4-butadiene (Merck); 1,6-hexane diamine (Merck); 1,8-octane diamine (Merck) and ethylene oxide (purity 99%) chemicals were used.

All solvents used were fractionally distilled just before use.

The structures of the synthesized compounds were confirmed by infrared (IR), nuclear magnetic resonance (^{1}H NMR).

Infrared absorption spectroscopy

The qualitative infrared absorption spectra of the synthesized compounds were recorded on a Beckman 4220 spectrophotometer.

Nuclear magnetic resonance (^{1}HNMR)

The ^{1}HNMR spectra of the compounds under investigation have been recorded in deuterated chloroform ($CDCl_3$) and /or in dimethyl sulphoxide (DMSO) as a solvent and tetramethyl silane (TMS) as an internal reference with 90 MHz signal and 4.000 gauss magnetic field.

Preparation of nonionic surfactants (II – V)a-c, were completed in the following reactions steps according to Scheme 1.

Preparation of dodecyl benzene sulphonyl chloride

0.01Mole dodecyl benzene sulphonic acid and 0.15 mole (17.73 g) thionyl chloride were added with few drops of pyridine into round bottom flask; the reflux system was filtered and the system was heated at 60 ^{o}C for 2 hr, the excess of thionyl chloride was removed under vacuum and the crude dodecyl sulphonyl chloride (1) was separated, washed twice with petroleum-ether 60-80 C^0 to give the product as yellow matter in good yield (95%) which is used in the second step without purification.

Preparation of N (2-hydroxyethyl) alkylbenzene sulphonamide (Ia-d)

0.01 Mole of 1 (3.45 g), 0.01 mole of alkylene diamine, 50 ml of dry benzene and drops of pyridine were stirred under reflux condenser for 2hr, cooled, poured on to acidified ice/water. The products were extracted with chloroform or petroleum ether and the solvent was evaporated, the crude products (Ia-d) were obtained. The reaction molar ratios and the products characterization were cited in Table 1.

TABLE 1. Reaction condition and product characterization of (Ia-d) .

Compd.	**Dodecyl benzene sulphonyl chloride g. mole**	**Alkylene diamine g. mole**	**Product. g. mole**	**Yield %**
Ia	(0.01, 3.45)	Ethylene diamine (0.01, 0.60)	Light brown cream (0.009, 3.4)	94
Ib	(0.01, 3.45)	Butylene diamine (0.01, 0.88)	Pale brown cream (0.0092, 3.6)	93
Ic	(0.01, 3.45)	Hexylene diamine (0.01, 1.16)	Yellow white Cream (0.00913, 3.8)	91
Id	(0.01, 3.45)	Octylene diamine (0.01, 1.44)	Yellow white cream (0.009, 4.0)	90

Ethoxylation

Compounds (Ia-d) were subjected to ethoxylation in the presence of basic catalyst (KOH), the general procedure of ethyoxylation was carried out in a semi micro apparatus (7) (Fig. 1) based on the measurement of volumetric flow of ethylene oxide (EO) before and after the reactor 7. the reactor has to be oxygen-free, because of softy and quality requirement (8,9). This was ensured by a preliminary flushing of the system with nitrogen. Nitrogen streams from bomb 1; EO flows from bomb 2 into three-way valve 3. The gas flows through buffer vessels 4 and 5 and a capillary flow meter 6 (differential monometer) into the reactor 7, where bubbles flow through the substrate and mixing the reaction mixture, while the reminder (not reacted) leaves the system through a buffer vessel 8 and capillary flow meter 9.

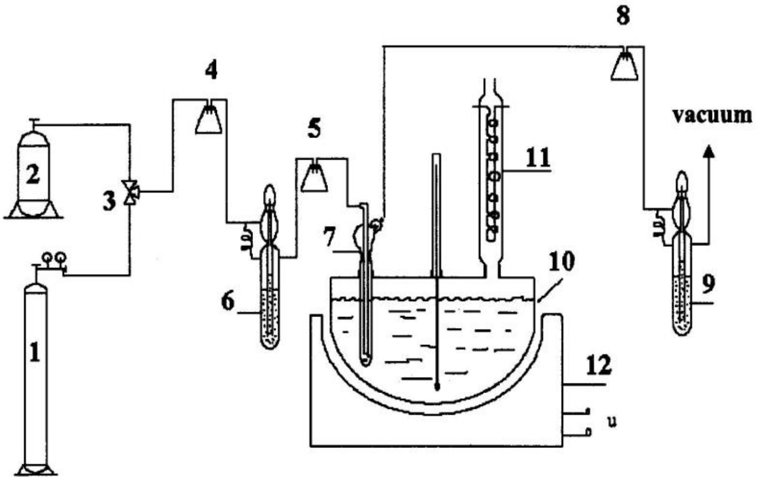


Fig.1. Apparatus of the hydroxyethylation (1.N2,2. ethylene oxide, 3. valve, 4,5,8. buffers, 6,9. differential manometers, 7. semimicro reactor, 10. boiling bath, 11. cooler, 12. heater).

Isothermal conditions for the reaction were ensured by a boiling liquid bath 10 (xylene at 140 ^{o}C) or with help of thermostat 12 at lower temperature. In the reactor tube the catalyst (KOH) was added to the substrate (Ia -d) (0.02 mole). The reaction mixture was heated to the desired temperature (140 ^{o}C). The system was purged with nitrogen to avoid oxygen (the explosion mixture of EO with air is 3-100 v %) (10) and to remove the water content of the reaction mixture in the case of alkali hydroxide catalyst. After 20 min, the gas stream was changed to EO. The EO gas added at a constant flow rate to produce the desired average degree of ethoxylation (ADE) (11) . Finally, the system of gas was changed to nitrogen to purge out the unreacted EO. The EO uptake (the average degree of ethoxylation) was determined from the increase of the mass of the reaction mixture and was controlled by spectroscopic tools (11). The ethoxylation reactions parameters are cited in Table 2 .

TABLE 2. Ethoxylation parameters of different substrates .

Starting substrate	**N**	**Catalyst mole%**	**Reaction temperature ^{o}C**	**ADE Mole / substrate**
2 (0.02 mole)	2	KOH (0.01 mole)	140 ^{o}C	5, 7 and 9
3 (0.02 mole)	4			
4 (0.02 mole)	6			
5 (0.02 mole)	8			

ADE Average degree of ethoxylation
n: number of CH_2 group

Estimation of polyethylene glycol contents

It was completed according to the Weibulls analytical method (12): 10 g of hydroxylated sample was distributed 3 times with equal amounts 5 M NaCl solution and ethyl acetate (100 ml), the polyethylene glycol (PEG) was in the aqueous layer (NaCl solution) and a pure hydroxyethylated sample in the organic layer. The organic solvent was distilled. The pure hydroxyethylated sample was weighted (w_1). The aqueous layer was extracted with 50 ml chloroform (3 times), after distillation of chloroform the PEG was weighted (w_2). The PEG percentage was calculated:

$$\text{PEG \%} = (w_2 /10) \times 100$$

Surface active properties of prepared nonionic surfactants

All these products were completed under neutral conditions in aqueous phase using oily and semisolid (cream matter) prepared at room temperature.

Surface and interfacial tensions

They were measured at 0.1 wt % aqueous solution at room temperature (25 ^{o}C) by using DU-NOUY tension meter (KRUSS type 8451) (13), also the interfacial tension was measured using parafine oil .

Cloud point

The cloud point, measure as inverse solubility characteristic of nonionic surface active agents, was determined by gradual heating 1% solution in a controlled temperature bath and recording the temperature at which the clear or nearly clear solutions become definitely turbid. Cooling the solutions until they become clear again checked the reproducibility of this temperature (14).

Wetting time

Wetting power of the tested surfactants was determined by immersing a sample of cotton fabric in 1.0 wt % aqueous solution of the surfactants and measuring the sinking time in seconds (15).

Foaming properties

Foaming properties were measured by Domingo I. F. and H. Meijer method (16) . The foam production for 1.0 wt % solution was measured by the foam height initially produced .

Emulsion stability

The emulsion was prepared from 10 ml of a 20 m mole aqueous solution of surfactant and 5ml of toluene at 40°C. The emulsifying properties were determined by the time, they took for an aqueous volume separating from the emulsion layer to reach 9ml counting from the moment of the cession shaking (17).

Biodegradability

Die-away tests in river water were performed employing the surface tension method (18) using a Du Notiy Tensiometer (Kruss Type 8451). Samples taken daily were filtered through No. 1 Whatman filter paper before measuring the surface tension. Biodegradation was calculated by the following equation:

$$D = \gamma_t - \gamma_0 / \gamma_{bt} - \gamma_0$$

(γ_t: surface tension at time t; γ_0: surface tension at time zero (initial surface tension); γ_{bt}: surface tension of the control sample at time t).

Biological Activity

Antibacterial and antifungal activities of the synthesized compounds

The well-plate methods (diffusion methods) were followed with some modification:

i. The spore suspension of the test organisms (gram negative bacteria-*Escherichia coil*-, gram positive bacteria *Staphylococcus aureus*-, yeast-

Candida albicans - and fungi- *Aspergillus niger-)* was prepared in previously sterile and checked inoculating flasks, each contained 50 ml of previously and checked medium of the following composition for the target test organisms as the following: For bacteria, nutrient broth medium: Beef extract 3.0 g, pepton 10.0 g and H_2O 1000 ml. For yeast and fungi, Czapeks Dox broth medium: Sucrose 30.0 g, $MgSO_4$ 0.5 g, KCl 0.5 g, $FeSO_4$ 0.01 g, $NaNO_3$ 3.0 g, K_2HPO_4 1.0 g and H_2O 1000 ml.

ii. A solid medium containing the following ingredients for bacteria (g/l) (beef extract 3.0 gs, peptone 10.0 g, agar 20.0 g, H_2O 1000 ml), and for yeast and fungi (sucrose 30.0 g, $MgSO_4$ 0.5 g, KCl 0.5 g, $FeSO_4$ 0.01 g, $NaNO_3$ 3.0 g, K_2HPO_4 1.0 g, agar 20.0 g, H_2O 1000 ml.) were sterilized and divided while warm (50: 55 ^oC) in 15 ml portions among sterile petri-dishes of 9 cm diameters.

iii. 0.2 Ml of the spore suspension was surface placed on the surface of the solid medium in the petri-dish and spread on all the surface.

iv. The test substances were dissolved in sterile water to make a solution of different concentrations (0.25%, 0.5%, 1%, and 2%).

v. In a petri-dish containing the culture of one of the chosen microorganisms, one well of 6 mm diameter was in the center of the dish. It was marked on the dish, and filled with the solution of the test substances with different concentrations.

vi. The petri-dishes were incubated at 5^oC for 2 hr to permit good diffusion and then transferred to an incubator at 37^oC for 48 hr for bacterial test organisms, and at 30^oC for 72 hr for yeast and fungi test organisms.

vii. Antimicrobial activities were observed after 48 hr of incubation time for bacteria at 37^oC, and after 72 hr of incubation time for yeast and fungi at 30^oC. The zones of inhibition were measured in mm (19) .

Results and Discussion

Preparation and structure elucidation

Dodecylbenzene sulphonic acid was used as a starting material in versatile synthesis of a series of nonionic surfactants as followes. At first, it was reacted with thionyl chloride in the presence of pyridine as a catalyst to produce dodecyl benzene sulphonyl chloride, which was confirmed via IR, which shows νSO_2 at 1050 cm^{-1}, $\nu_{C\text{-}H\ aromatic}$ at 3050 cm^{-1} and $\nu_{C\text{-}H\ aliphatic}$ strain in region of (2922-2854) cm^{-1}, and ^1H NMR spectra which shows the following signals: 0.96 (t, 3H, terminal CH_3), 1.29-1.62 (m, 20H, OCH_2 of alkyl chain) and 7.44-7.88 (m, 4H, ArH).

N-(3-amino alkyl)-4-dodecylbenzene sulphonamide (Ia-d) were prepared by the reaction of dodecyl sulphonyl chloride with alkylenediamine in the presence of pyridine as a catalyst. The compounds (Ia-d) were confirmed via IR and ^1H NMR spectra.

IR spectra show νNH, NH_2 in region of (3230-3400), νSO_2 at 1070 cm^{-1}, $\nu_{C\text{-}H\ aromatic}$ at 3010 cm^{-1} and $\nu_{C\text{-}H\ aliphatic}$ of alkyl chain in region of (2920-2850) cm^{-1}.

^1H NMR of (Ib) shows signals: 0.9 (t, 3H, terminal CH_3), 1.2-1.5 (m, 20H, 10 CH_2 of alkyl chain), 2.3-2.7 (m, 12H, 6CH_2 of diamine), 4.3 (3H, NH and NH_2) and 7.44-7.88 (m, 4H, ArH).

Ethoxylation of compounds (Ia-d) with different average degree of ethoxylation (5, 7 and 9 moles) gave nonionic surfactants (II-Va-c).

Surface active properties of the prepared nonionic surfactants

The surface active and related properties, including surface and interfacial tension, cloud point, foaming power, wetting time, and emulsification properties were investigated to evaluate the possible application of these products in the different industrial fields.

Surface and interfacial tension

In comparing surfactants or emulsifiers by surface or interfacial tension measurements, two factors, efficiency and effectiveness need to be considered. Ahmed(20) suggests that a good measure of efficiency is the amount of a surfactant required to reduce the tension by 20-dyne/cm and the minimum tension obtainable with the surfactant measured its effectiveness. The values of surface and interfacial tension of the synthesized products increased by increasing the number of ethoxy group per molecule of products as shown in Tables 3-6.

Cloud point

All the synthesized products have high cloud points, which gave the good performance in hot water. Generally, the cloud point increases with increasing the number of ethoxy group per hydrophobic molecule (20) in the prepared samples . The cloud points are higher than 100 (Tables 3-6).

Wetting time

All the products show decreasing in wetting time, where good wetting times are recorded with a low ethylene oxide content (21) as in Tables 3-6.

Foaming height

In general, the nonionic surfactants form unstable foam. It was reported that the foaming height of the prepared surfactants increases with increasing of both ethylene oxide unit and hydrophobic part per molecule of surfactant (21) as in Tables 3-6.

Emulsifying properties

Emulsion stability was measured using standard procedures. From the data, the emulsifying properties increase with decreasing number of ethylene oxide units and increase with increasing of alkyl chain (22) (*c.f* . Tables 3-6).

TABLE 3. Surface properties of the ethoxylated synthesized surfactants.

Compd	n	x + y + z	S. T 0.1wt.% dyne/cm	I.F.T 0.1wt.% dyne/cm	Cloud point 1.0 wt.% ^{o}C	Foam height 1.0 wt. % (mm)	Wetting t 0.1% sec.	Emul.S. 20 mmole: min. sec
						0^{o} mm		
IIa	2	5	40	16	>100	180	40	11
IIb		7	41	18	>100	200	45	10
IIc		9	41.5	17	>100	215	50	9

TABLE 4. Surface properties of the ethoxylated synthesized surfactants.

Compd	n	x + y + z	S. T 0.1wt.% dyne/cm	I.F.T 0.1wt.% dyne/cm	Cloud point 1.0 wt.% ^{o}C	Foam height 1.0 wt. % (mm)	Wetting t 0.1% sec.	Emul.S. 20 mmole: min. sec
						0^{o} mm		
IIIa	4	5	40.5	15	>100	200	45	12
IIIb		7	41.5	16	>100	220	50	11
IIIc		9	43	17	>100	230	50	10

TABLE 5. Surface properties of the ethoxylated synthesized surfactants.

Compd	n	x + y + z	S. T 0.1wt.% dyne/cm	I.F.T 0.1wt.% dyne/cm	Cloud point 1.0 wt.% ^{o}C	Foam height 1.0 wt. % (mm)	Wetting t 0.1% sec.	Emul.S. 20 mmole: min. sec
						0^{o} mm		
Iva	6	5	41	14	>100	210	60	15
IVb		7	42	15	>100	225	65	14
IVc		9	44	16	>100	240	70	13

TABLE 6. Surface properties of the ethoxylated synthesized surfactants.

Compd	n	x + y + z	S. T 0.1wt.% dyne/cm	I.F.T 0.1wt.% dyne/cm	Cloud point 1.0 wt.% ^{o}C	Foam height 1.0 wt. % (mm)	Wetting t 0.1% sec.	Emul.S. 20 mmole: min. sec
						0^{o} mm		
Va	8	5	41.5	16	>100	200	80	17
Vb		7	43	16.5	>100	230	90	16
Vc		9	45	17	>100	250	100	15

n = number of methylene groups; x + y + z = moles of EO.

Biodegradability

A biodegradation die-away test in ordinary river water gave satisfactory results (Tables 7). Each experiment was repeated at least three times, and the results are reported as averages of three values.

The biodegradation was expressed by measurement of the surface tension with time (day). The rate of degradation of these compounds depends on the size of molecule; bulky molecule diffuses through the cell membrane and its degradation is more difficult, this means that these compounds with lower moles of ethylene oxide are more degradable than that which contains higher moles of ethylene oxide (23) In general, the products have a much higher rate of degradation ranging about 95% degradation during around 6-7 days. It could also be shown that the biodegradation of the surfactants decreased with increasing number of carbon atoms of the alkyl chain.

TABLE 7. Biodegradability of the prepared nonionic surfactants.

Compd.	**N**	**N**	**1st day**	**2nd day**	**3rd day**	**4th day**	**5th day**	**6th day**	**7th day**
IIa	5	2	60	69	78	87	95	-	-
IIb	7		58	66	75	83	92	99	-
IIc	9		57	64	72	80	88	93	-
IIIa	5	4	58	65	73	82	93	98	-
IIIb	7		55	63	71	81	90	96	-
IIIc	9		53	60	68	75	83	90	-
Iva	5	6	54	62	69	77	83	88	-
IVb	7		52	60	66	72	80	86	-
IVc	9		50	58	65	70	78	82	-
Va	5	8	53	60	68	75	81	86	-
Vb	7		51	58	65	72	79	84	-
Vc	9		48	56	63	69	75	80	-

n: number of mole of EO; n: number of methylene group.

Biological activity

All the compounds prepared were screened for their activity against Gram-positive bacteria (*Stophyloccus aureus, Bacillus subtilis, Bacillus cereus*), Gram-negative bacteria (*Pseudomonas aurignosa, Echerichia coli, Enterobacter aerogenes*), as well as fungi (*Aspergillus niger, Penicillium italicum, Fusarium oxysporum*). The results are listed in Tables 8 and 9.

TABLE 8. Antibacterial activity of the prepared compounds.

Compds.	*Stophyloccus aureus s*	*Bacillus subtilis*	*Bacillus cereus*	*Pseudomonas aurignosa*	*Escherichia coli*	*Enterobacter aerogenes*
IIa	7	10	9	8	11	7
IIb	12	14	13	11	12	12
IIc	9	11	13	8	13	9
IIIa	8	8	11	9	13	11
IIIb	7	9	7	7	10	13
IIIc	12	10	13	12	13	11
Iva	12	9	10	11	8	9
IVb	11	12	10	9	9	10
IVc	14	13	12	12	12	13
Va	12	11	12	13	13	7
Vb	13	11	8	10	10	9
Vc	9	12	10	11	11	10
Amoxicillin	13	10	12	12	11	10

TABLE 9. Antifungal activity of the prepared compounds.

Compds.	*Aspergillus niger*	*Penicillium italicum*	*Fusarium oxysporum*
Iia	8	7	8
Iib	10	8	7
Iic	13	10	9
IIIa	9	11	8
IIIb	10	9	7
IIIc	12	7	9
Iva	9	10	11
Ivb	11	11	11
Ivc	13	9	10
Va	11	11	9
Vb	10	9	10
Vc	12	12	11
Amoxicillin	13	9	8

It is apparent from the data listed in Table 8 that some of the synthesized compounds showed antibacterial activity. However, concerning the activity against Gram-positive bacteria *(Bacillus subtilis)*, compound IIb showed excellent activity, compounds IVb, IVc, exhibit good activity, whereas compounds, IIa, IIIb both showed low activities. On the other hand, the Gram-negative bacteria *(Pseudomonas aurignosa)* showed high responses to five of the prepared products. Compound IVc showed the maximum activity, higher than that of amoxicillin. Compound Vc exhibits excellent antibacterial activity towards *Entrobacter aerogenes.*

Concerning the data of antifungal activity in Table 9, compounds IIc, IVc showed excellent activity against *Aspergillus niger,* while compounds IVb, Vc exhibit good activity. Also, compound IVa displays excellent activity toward *Penicillium italicum*. In general, the data obtained from the microbiological screening showed that the activities of some synthesized compounds are equal to and sometimes greater than those of the reference drugs used(24) .

Conclusion

Dodecyl benzene sulphonic acid is one of the most popular and cheap crude material used in the synthesis of a large number of surface active agents. The synthesized compounds containing sulphonamide group have good biological activities cooperated with surface and are environment-friendly**.**

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تحضير منظفات غير أيونية بإستخدام مشتقات بترولية

كوثر عبد الحليم حبيش ، محمد حسين و جمال الهادى
قسم الكيمياء – كلية العلوم – جامعة بنها – بنها – مصر .

تم في هذا البحث تحضير سلسلة من المركبات من a-c (II - V) كمنظفات غير أيونية وذلك بإستخدام بعض مشتقات البترول الشائعة و الرخيصة الثمن (دوديسيل بنزين صلفونيك أسيد) . و ذلك بتفاعل هذا المركب مع صيونيل كلوريد يتبعها سلسلة من تفاعله مع ألكيلين داي أمين (n =8,6,4,2) ليعطي (Ia-d) على التوالي .

ثم تم أخذ هذه النواتج لتتفاعل مع إيثلين أوكسيد لينتج المنظف المطلوب و هو منظف غير أيوني a-c (II-V) وأكدت هذه المركبات بواسطة التحاليل (HNMR&IR) دراسة أطياف الأمتصاص للأشعة تحت الحمراء و الرنين النووي المغناطيسي و بالإضافة إلى ذلك فقد تم قياس الخواص السطحية لهذه المركبات، أختبار قدرتها على التحلل البيولوجي و قد وجد أن لها نشاط سطحي جيدا، كذلك لها القدرة على التحلل البيولوجي السريع مما يجعلها مركبات ذات نشاط سطحي آمن و غير ملوثة للبيئة.