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Improved Manual Dishwashing Liquid Detergent Compared to that Produced by Multinational Companies in Egyptian Market

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IN THIS study, formulation of manual dishwashing liquid detergent with ionic, nonionic and zwitterionic surfactants mixtures were investigated in respect to viscosity, cloud point, foaming power, foam features, washing performance, irritation test, in addition to its cost, in comparison with that produced by multinational companies in their own factories in Egypt. It has been found that the formulations containing Linear alkyl benzene sulphonic acid are characterized by higher performance of removing stain, without any stain deposition on dishes, in addition to moderate foaming power which is diminished with hard water. Our proposed formulations 10% Sodium alkylbenzene sulfonate with 2% sodium laurylether sulphate and 2% coconut diethanolamide or 2% Cocamidopropylamine Oxide have resulted in better washing performances, less irritations, higher viscosity, comparable cloud points and lower cost than multinational market products H and P which contain 12% Sodium linear alkylbenzene sulfonate with 6% sodium laurylether sulphate and 9% sodium laurylether sulphate and 6% Cocamidopropylamine oxide respectively.

Keywords: Manual dishwashing liquid detergent, Sodium linear alkylbenzene sulfonate, Irritation test, Cloud point, Foaming power.

Introduction

Manual dishwashing liquid detergents (MDWL) has still been of considerable importance in the market, and are used in almost every household, even in parallel with automatic dishwashers. The Modern definition of MDWL is aqueous solution of different surfactants mixture, combined with other specific materials whose function is to increase foam, stabilizing and homogenized the formulation, and provide the right viscosity [1]. Manual dishwashing liquid detergents (MDWL) represents the second largest products category in the Middle East with total consumption estimated around 700,000 tons per year. Per captia consumption ranges from 1 kg in Egypt to 3 kg in Iran and Saudi Arabia [2]. Nowadays, dishwashing liquid detergents often contain certain mixtures of different types of surfactants to strengthen their cleaning performance, and the ability to remain mild to the hand skin [3]. The types and level of surfactants control the basic parameters of dishwashing liquid detergent, like foaming, cleaning, washing performance and viscosity [4]. Mixtures of different surfactants have many industrial applications because they show better characteristics than their building units, these features can be explained by synergistic interactions among building units in mixed micelles [5-8].

Materials

The surfactants used in this study are shown below. These were commercial samples and were not specially purified.

1) Linear alkyl benzene sulphonic acid C11-C13 (LABSA). Assay: 96 - 97.5%,
2) Sodium laurylether sulphate C12-C14 2EO (SLES-2EO). Assay: 69%,
3) Cocamidopropylamine Oxide (CAO). Assay: 29%.

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4) Coconut diethanolamide (CDEA). Assay: 90%.
5) Cocamidopropylbetain (CAPB). Assay: 28-32%.
6) Citric Acid 10% W/w solution and sodium hydroxide 10 % W/w solution were used for adjustment of pH values.
7) Two manual dishwashing liquid detergents from local market produced by two multinational companies, in their own factories in Egypt. The First (Product H) has around 23 % market share, and the second one (Product P) has around 12% market share.

Methods

Formulation preparation

Formulations were prepared in 500 g quantity, in which ingredients were mixed in the following sequence: water, sodium hydroxide, linear alkyl benzene sulphonic acid C11-C13 , then pH was adjusted to 9 -10 by citric Acid 10% solution and/or sodium hydroxide 10 % solution, then sodium lauryl ether sulphate C12-C14 2EO and/or cocamidopropylamine oxide and/or coconut diethanolamide and/or cocamidopropylbetain were added. Finally, pH was adjusted to 7–8 by adding citric Acid 10% solution and/or sodium hydroxide 10 % solution as required[9].

Measuring of pH values

pH degrees of formulations were measured for 0.1% solution at 25 ±2.0 °C using Jenway model 3510[10].

Measuring of viscosity

The apparent viscosities of formulations was measured using Brookfield DV–E viscometer LVDV-E with Spindle number 6 in 250 ml container at temperature 20 ±1.0°C [11].

Measurement of cloud point

Fifty milliliters of formulations was poured into test tubes and a thermometer was immersed in each formulation's tube and then test tubes were immersed in -3°C chillers. The temperatures reading was recorded when the formulations in test tube started to turn cloudy [12].

Measurement of surfactants content

Anionic-active matters hydrolysable and non-hydrolysable were determined under acidic conditions. Titration of an aliquot of a sample solution with benzethonium chloride solution according to the direct two- phase titration procedure specified in ISO 2271:1989 [13] was done. Hydrolysis, by refluxing under acidic conditions, of a second aliquot of the sample solution was carried out. Calculation of the contents of hydrolysable and non-hydrolysable anionic-active matter as per ISO 2870:2009 [14] were recorded. Nonionic surfactants were measured after treating an alcoholic extraction aliquot of a sample solution with anionic and cationic resin then dried out and weighted. Obtained results from analysis were prepared as batch then its forming power and washing performance in comparison were recorded with market samples to confirm their compositions.

Measurement of foaming power

Two hundred milliliters of 1 % formulations solution at 30 °C was freely dropped from separating funnel into a graduated cylinder. Separating funnel was placed in such way so that its outlet tube points towards the center of the graduated cylinder and the distance between its lower edge and the graduated cylinder was five centimeter. Initial volume of foam generated was recorded and after 30 min foam volume was recorded again [15,16].

Measurement of washing performance.

Soiled dinner plates were washed by mechanical rotating motion in 0.1% solution of prepared formulations under standardization conditions until the last point, when the disappearance of the foam was reached the number of plates washed was recorded, and also the number of stained plates was recorded if any [17].

Irritation test

This test was carried out by CHELAB srl [18,19] which is a private and independent laboratory (www.chelab.it). The procedure was followed by CHELAB srl as follow. The skin under investigation was cleaned with ethyl alcohol 70%, it is necessary to apply blasters on paravertebral area, at scapular level, or on the forearm to be let on site for 24 hours. The volunteer was reminded not to wash the area during the whole test. After this interval of time, blasters were removed by a dermatologist, who would clean the specific areas from all eventual product residues after 15 minutes, this period of time is necessary to extinguish blaster irritation; the dermatologist performs a first assessment of the eventual irritation and after 24 hours from blaster removal dermatologist carries out the second assessment.

Result and Discussion

Effect of sodium chloride on viscosities and cloud points of Manual dishwashing liquid detergent formulations.

Table 1 shows that the viscosity and cloud point of 10% LABSA formulations were increased by the addition of sodium chloride. Viscosity reached to 1150 millipascal-second (mPa.s) and cloud point recorded at 15 °C with 0.6% sodium chloride, any further addition of sodium chloride leads to decreasing in viscosities and increasing of cloud point.

In formulation of 2% SLES-2EO and 10% LABSA; the ability of formula to absorb more sodium chloride had been improved but viscosities were lower than in case of 10% LABSA formulation and cloud point was higher. The addition of 2% CAPB to 10% LABSA formulation was the best option for achieving high viscosities with zero cloud point. Addition of 0.15% Sodium chloride increased the viscosity to 5800 mPa.s and zero cloud point. Formulation of 2% CAO with 10% LABSA was given lower viscosities than formation of 2% CAPB with 10% LABSA and higher cloud points. CDEA (2%) and 10% LABSA formulation was not compatible with sodium chloride, whereas cloud points increased with low viscosities by addition of sodium chloride.

LABSA/ SLES-2EO and LABSA/CDEA formulations which are very sensitive for sodium chloride and caused cloudy and low viscosities. LABSA/CAPB and LABSA/ CAO were more compatible with sodium chloride which leads to achieving the desired viscosity to consumer even in case of being of low percentage. Peak points at which viscosities started to decrease were

TABLE 1. Effect of Sodium chloride on viscosities and cloud points of different manual dishwashing liquid detergent.

LABSA 10%.								
% Sodium chloride	0	0.2	0.3	0.4	0.6	0.8	0.9	1
Viscosity mPa.s	90	250	370	480	580	1150	750	500
Cloud point. °C	0	0	0	0	11	15	20	25
10% LABSA % + 2% SLES-2EO								
% Sodium chloride	0	0.6	0.7	0.8	0.9	1	1.1	1.2
Viscosity mPa.s	70	80	120	180	195	250	380	410
Cloud point. °C	0	0	0	2	6	7	12	15
10 % LABSA + 2% CAPB.								
% Sodium chloride.	0	0.03	0.05	0.07	0.09	0.1	0.15	0.3
Viscosity mPa.s	590	1840	2350	3750	4100	4500	5800	3500
Cloud point. °C	0	0	0	0	0	0	0	3
10 % LABSA+ 2% CAO.								
% Sodium chloride	0	0.3	0.5	0.6	0.8	0.9	1	1.2
Viscosity mPa.s	750	530	750	1150	1700	2700	3800	1050
Cloud point. °C	0	0	0	0	0	2	5	23
10 % LABSA + 2% CDEA.								
% Sodium chloride	0	0.3	0.5	0.8	1.2	1.8	2	2.2
Viscosity mPa.s	60	120	200	370	800	1600	1300	760
Cloud point. °C	0	0	0	0	6	15	20	25

TABLE 2. Analysis results of two multinational companies' local products of manual dishwashing liquid detergent H and P.

Product code	**LABSA%.**	**SLES-2EO%.**	**CAPB%.**	**CAO%.**	**pH**	**Cloud point °C**	**Viscosity mPa.s**
Product H	12	6	0	0	6.8	Less than 0 C.	4100
Product P	0	9	0	6	7.1	Less than 0 C.	3650

recorded with all formulation with increasing sodium chloride content except 10% LABSA % plus 2% SLES-2EO formulation.

Katarzyna, et al. [20] reported that negative effects of sodium chloride have been noticed in two points: Firstly, that Critical Micelle Concentration (CMC) increases with increasing ionic strength, secondly, foam height decreases with increasing salt concentration.

Effect of surfactants mixtures and its concentration on viscosities and cloud points of manual dishwashing liquid detergent and comparing with multinational companies' local products

In this study, we selected some manual dishwashing liquid detergent (MDWL) formulations which would give viscosity in limit of our multinational samples without needing to use any kind of thickening agent. We consider thickening agent is an extra cost without any positive effect on product performances. Our measurements of viscosities, cloud points, surfactants composition and pH of Local markets products H and P were shown in Table 2. Results show that viscosities in range of 3650 – 4100 mPa.s and cloud point always below 3°C. These tables were used during our study as benchmark for viscosities rang preferred by Egyptian customer.

A. As shown in Table 3, an increase in concentration of LABSA in formulation did not contain another type of surfactant caused small increase in viscosity, and a notable increase of cloud point of formulations (Table 3). At 12% of LABSA formulation's cloud point raised to 3°C with very low viscosity 190 mPa.s and at 20% of LABSA; viscosity increased to 2100 mPa.s with cloud point 21°C. This formulation gave viscosity lower than customer preferred range and cloud point higher than what needed for stable product. We notice that; viscosity of 10% LABSA was very low (80 mPa.s) and cloud point is zero, so we chose it as a base formulation to develop the best surfactants mixture so as to come up with preferred viscosity and cloud point to consumer with low cost.

TABLE 3. Relation between % of linear alkyl benzene sulphonic acid C11-C13 (LABSA) with its viscosities and cloud points.

LABSA %	8	10	12	14	16	18	20
Viscosity mPa.s	50	90	190	260	850	1300	2100
Cloud point. °C	0	0	3	7	13	17	21

B. As shown in Table 4, addition of SLES-2EO with different concentration up to 10% to 10% LABAS formulation did not affect strongly on viscosity, which increased to 320 mPa.s and cloud point remained zero. Continuous increasing of SLES-2EO caused a notable increase in viscosities with cloud point remained zero. Viscosity and cloud point of 18% LABSA was higher than formulation of 10% LABSA and 8% SLES-2EO.

C. Additions of CAPB with different concentration up to 1.8% to 10% LABSA formulation had slightly effect on viscosity which was still lower than consumer preferable value. Continuous increasing of CAPB caused a large increase in viscosity; unfortunately cloud point increased as well. Only 0.1% CAPB changed the formulation viscosity and cloud point dramatically .e.g. 2.2 % of CAPB gave very low viscosity (880 mPa.s) and low cloud point also (0°C) but 2.3% of CAPB gave high viscosity even higher than consumer preferred level (8700 mPa.s) but cloud point was also high (10 °C). So careful addition of CAPB should be followed to achieve the desired viscosity with LABSA formulation. Viscosities of 3.3% CAPB with 10% LABSA formulation decreased again as result of salting out phenomena.

D. CAO had the same behavior manner of CAPB but with higher dosage. e.g CAO (4.2%) presenting comparable results to 2.3% of CAPB with higher cloud point. Viscosities of CAO with 10% LABSA formulation decreased again at 8% of CAPB to be 10700 mPa.s and cloud point 18 C.

E. Addition of CDEA with different concentration up to 5.5% to10% LABSA formulation didn't have a strong effect on viscosity and cloud point (700 mPa.s zero cloud point). Continuous increasing of CDEA had resulted in a high increase in viscosity with frequent zero cloud point.

TABLE 4. Viscosities and cloud points of 10% linear alkyl benzene sulphonic acid formulation and sodium laurylether sulphate (SLES-2EO) or cocamidopropylbetain (CAO) or cocamidopropylamine oxide (CAPB) or coconut diethanolamide (CDEA) with different percentages.

LABSA%	**10**							
Viscosity mPa.s	90							
Cloud point. °C	0							
10% linear alkyl benzene sulphonic acid and sodium laurylether sulphate (SLES-2EO) formulations.								
Addition of SLES-2EO %	2	4	8	12	16	20	24	26
Viscosity mPa.s	70	100	320	650	980	4800	8750	12300
Cloud point. °C	0	0	0	0	0	0	0	0
10% linear alkyl benzene sulphonic acid and cocamidopropylamine oxide (CAPB) formulations,								
Addition of CAPB %	0.55	1.1	1.65	2.2	2.3	2.75	3.3	3.85
Viscosity mPa.s	25	65	85	880	8700	24000	800	600
Cloud point. °C	0	0	0	0	10	18	10	8
10% linear alkyl benzene sulphonic acid and cocamidopropylbetain (CAO) formulations.								
Addition of CAO %.	0.6	1.2	1.8	2.4	3	3.6	4.2	4.8
Viscosity mPa.s	20	180	230	1020	3350	6600	8900	12200
Cloud point. °C	0	0	0	11	14	18	21	29
10% linear alkyl benzene sulphonic acid and coconut diethanolamide (CDEA) formulations.								
Addition of CDEA%.	5	5.5	6	6.5	7	7.5	8	8.5
Viscosity mPa.s	300	700	1300	4300	10300	14700	15700	21340
Cloud point. °C	0	0	0	0	0	0	0	0

SLES and CDEA were the best additives for 10% LABSA formulation to increase its viscosities without increasing of cloud point. 20% SLES-2EO or 6.5% of CDEA were needed to achieve the desired level of viscosity 4300 mPa.s with zero cloud point, but their formulation was very high cost. Economic formulation of 10% LABSA formulation was achieved with CAPB % between 2.2 to 2.3 or CAO %3 to 3.2%.

Foaming power of manual dishwashing liquid detergent proposed formulations and local products of multinational companies under different levels of water hardness

We studied the ability of proposed formulation to generate foaming and foam stability with time. Because foam generation is extremely important criteria for customer to evaluating MDWL [21]. SLES-2EO has very effective change in foaming power of 10 %LABSA as it increased the initial foaming level from 85 ml in case of 10 %LABSA alone to 115 ml and increased the foam level after 30 min from 35ml to reach 80 ml (Table 5). SLES-2EO also improved the foaming level in hard water to became 75 ml with 500 ppm water instead of 20 ml. in case of 10 %LABSA alone. The second best was CAO [22] followed by CAPB. Product P has a very high foaming power because it contains a high percentage of SLES-2EO and CAO. Slightly improving were noticed in initial foaming power of 10 % LABSA formulations while adding of CDEA with soft water, but it increased the foaming stability by time. CDEA improved foaming power with hard water but its effect was lower than SLES-2EO or CAO or CAPB [23,24].

TABLE 5. Intial and after 30 minutes foaming power of different manual dishwashing liquid detergent formulations in compare with local products of multinational companies at different levels of water hardness.

% Surfactant / water hardness.	200 ppm		500 ppm		1500 ppm.	
Time.	Initial Foaming power	Foaming power after 30 minutes	Foaming power Initial	Foaming power after 30 minutes	Foaming power Initial	Foaming power after 30 minutes
10%LABSA.	85	35	75	20	30	10
10% LABSA + 2% SLES-2EO.	115	80	90	75	55	40
10% LABSA + 2% % CAPB.	85	60	80	50	40	30
10% LABSA +2% CAO.	95	70	75	55	45	30
10% LABSA +2% CDEA.	90	60	75	55	40	30
10% LABSA + 2% SLES+2% % CAPB.	125	90	105	90	90	75
10% LABSA + 2% SLES+2% CAO.	130	90	105	90	90	75
Product H	120	80	90	75	55	40
Product P	145	100	125	95	100	85

The best foaming performance was achieved with the formulations of 10% LABSA plus 2% SLES-2EO plus 2% CAO (or 2% CAPB) with both of soft and hard water.

Washing performance of manual dishwashing liquid detergent proposed formulations and comparing with multinational companies' local products.

Evaluating washing performance ASTM method D4009/92 (2011) [25] depending mainly on observation of foaming levels, where the performance of dishwashing liquid was diminished when foaming disappeared. It is shown in Table 6 and 7 that SLES-2EO, CAPB and CAO had been improved the washing performance of formulations while CDEA did not affect at all with washing performance. Formulations that contain less than 10% LABSA have higher numbers of stained plates rather than that contain 10% LABS. e.g. 8% LABSA plus 2% SLES-2EO formulation had 3 washed plates until foam disappeared two of them had stain on it. In another case 10% LABSA plus 2% SLES-2EO formulation had 3 washed plates until foam disappeared where there was no any stain on it.

Manual dishwashing liquid detergents (MDWL) with 10% LABSA plus 2% SLES-2EO plus 2% CAO (or 2% CAPB) had nine washed plates until foam disappeared without any stain on it. Products H which contain 12% LABSA plus 6% SLES had five washed plates until foam disappeared without any stain on it. Products P which contain 9% SLES-2EO plus 6% CAO which had twelve washed plates until foam disappeared but nine plates from them had stain on it. That means that my Manual dishwashing liquid detergents formulations gave better results than that shown by product H and P which produced by two multinational companies, Cost of my Manual dishwashing liquid detergents formulations were lower than H and P product (Table 6).

E-Irritation test of different surfactants mixture formulations in compare with local products of multinational companies

Irritation test has shown that formulations that contain 10% LABSA with 2% SLES-2EO and 2% CAPB (or 2% CAO) hadn't caused any irritation to skin. Products H which contains 12% LABSA plus 6% SLES also has shown no irritation, whereas products P which contains 9% SLES-2EO plus 6% CAO caused slightly irritation to skin.

TABLE 6. Compositions of different proposal for manual dishwashing liquid detergent formulations in compare with local products of multinational companies H and P and its cost.

Proposal number	1	2	3	4	5	6	7	Product H	Product P
LABSA.	8	8	10	10	10	10	10	12	0
SLES-2EO.	0	2	2	0	0	2	2	6	9
CAPB.	0	0	0	0	0	2	0	0	0
CAO.	0	0	0	2	0	0	2	0	6
CDEA.	0	0	0	0	2	0	0	0	0
Cost euro 100 Kg	10.7	14.9	17.6	21.5	16.1	24.1	25.7	28.6	37.0

Conclusion

Finally, we might conclude the following points compared with manual dishwashing liquid detergent (MDWL) produced by multinational companies and our preparations.

1. LABSA improved the washing performances of MDWL especially it reduced the numbers of stained plates, avoided the dirt to stick again to plates.
2. CAPB and CAO supported by sodium chloride could increase viscosity of MDWL under careful controlling of their addition without need to add thickening agent.
3. SLES-2EO was strongest surfactant in concern of foaming power which also had high resistance to hard water.
4. We might avoid using high percentage of SLES-2EO or CAO which may cause skin irritations.
5. My prototypes formulation 10% LABSA with 2% SLES-2EO and 2% CAPB (or 2% CAO) had lower in cost than Market products.

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تركيبات محسنه للمنظفات السائلة الخاصه بغسيل الأطباق يدويا مقارنة بالمنظفات التي تنتجها الشركات العالميه في السوق المصري

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[١]قسم الزيوت و الدهون - المركز القومي للبحوث - شارع البحوث - الدقي - القاهرة - مصر.
[٢]قسم الكيمياء - كلية العلوم - جامعة عين شمس - القاهرة - مصر.

فى هذا البحث يتم دراسه خواص المواد ذات النشاط السطحي الانونيه واللانيونيه والمترددە وذلك حاله تواجدها فى تركيبات الصابون السائل الخاص بغسيل الاطباق بصوره مفرده او ثنائيه او ثلاثيه . يتم دراسه خواص المواد ذات النشاط السطحي فى التركيبات المختلفه وذلك من خلال قياس القدره على تكوين رغوه و مدى ثبات هذه الرغوه فى حاله استخدام ماء عسر و درجه لزوجه هذه التركيبات و تاثيرها على الجلد كذلك قدرتها التنظيفيه ومقارنتها بالمنظفات التي تنتجها الشركات العالميه في مصر.

نتائج الدراسه تشير الى ان التركيبات التى تحتوي على سلفونات ألكيل بنزين الصوديوم يتسم بالأداء العالي لإزالة البقع ، دون أي ترسب على الأطباق ، بالإضافة إلى قوة رغوة معتدلة تتناقص مع الماء العسر. التركيبات المقترحة ١٠٪ سلفونات ألكيل بنزين الصوديوم مع ٢٪ كبريتات الصوديوم لوريل ايثر و ٢٪ من دي ايثانول اميد جوز الهند أو ٢٪ من أكسيد الكوكاميد بروبيل امين قد نتج عن أداء أفضل للغسيل وأقل تهيج ولزوجة أعلى ونقاط تشبير مماثلة وتكلفة أقل من منتجات السوق متعددة الجنسيات H و P التي تحتوي على ١٢ ٪ سلفونات ألكيل بنزين الصوديوم مع ٦ ٪ كبريتات الصوديوم لوريلثير او ٩ ٪ كبريتات الصوديوم لوريل ايثر و ٦ ٪ أكسيد الكوكاميد بروبيل امين على التوالي.