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Synthesis, Identification and Surface Active Properties of Some Nonionic Surfactants Containing Quinazolinone Ring.

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Abstract: *This research includes synthesis of new heterocyclic derivatives of quinazolinone and studying the possibility for their application as surfactants. The preparation process started by the reaction of pentadecanoyl chloride with anthranilic acid gave 2-pentadecyl-4H-benzo[3,1]oxazin-4-one [1] which was used as starting material to synthesis some heterocyclic compounds by a reaction with nitrogen nucleophiles. Compound [1] as starting material reacted by three ways. The first way react Compound [1] with semicarbazide, thiosemicarbazide and phenylsemicarbazide in pyridine as solvent to obtained [1-(4-oxo-2-pentadecylquinazolin-3(4H)-yl) urea [2], thiourea [3], phenylthiourea [4] respectively]. The second way include the reaction of Compound [1] with 99% hydrazine hydrate to produce 3-amino-2-pentadecylquinazolin-4(3H)-one [5], then converted to amino derivatives [6-8] by the reaction of Compound [5] with different halogenated compounds such as (benzoyl chloride, acetyl chloride and chloro acetyl chloride) in DMF and pyridine as catalyst. The third way include the reaction of compound [1] with different substituted amine and amide compounds (thiourea, hydroxylamine hydrochloride, glycine, ammonium acetate and phenyl hydrazine) respectively to obtain derivatives of quinazoline [9-14]. The produced compounds [2,4,5,7 and 12] react with propylene oxide in different moles (n=3, 5 and7) to yield novel groups of nonionic compounds having a function as surface active agents. The surface active properties like surface tension and interfacial tension, wetting time, cloud point, emulsion stability, foam height and CMC of these compounds were measured and showed a pronounced surface activity, good emulsifying properties and highly foaming in some of these compounds. Newly synthesized compounds were identified via spectral methods; their [FTIR and some of them by ^{1}HNMR, ^{13}C-NMR] and measurements of some of its physical properties.*

Keywords: *Quinazolinone derivatives, surface active agent, nonionic surfactant,*

I. Introduction

Nonionic surfactant is one of the most important groups of surfactant with growing industrial interest and can by synthesized by the ethoxylation or propxylation (addition of ethylene oxide or propylene oxide) of hydrophobic organic compounds containing active hydrogen in the presence of catalyst such as KOH.[(1)]

Quinazolinone derivatives which may have pharmaceutical and industrial applications [(2)]. These derivatives are the subject of many research studies due to their widespread potential biological activities such as antimicrobial [(3)], anti-inflammatory [(4)], antitumor [(5)] anticancer [(6)] corrosion inhibitors [(7)] and anti-oxidant [(8)]. This led us to synthesis of novel group of nonionic surfactant containing this nucleus from long chain fatty acid (palmitic acid). Some of the synthesized compounds which have active hydrogen atom were subjected to react with propylene oxide by different moles (n=3, 5 and7) to produce nonionic compounds.

The main objective of this investigation is to synthesis and identification of new surface active agents containing quinazolinone nuclei and studies the surface active properties as surface tension and interfacial tension, wetting time, cloud point, emulsion stability, foam height and CMC.

II. Experimental

2.1 Materials and Instruments

Chemicals used in this work are supplied from BDH, Fluka, Merck and Sigma Aldrich companies and used without further purification. Melting points were uncorrected and registered via digital Stuart scientific SMP3 melting point device .Thin layer chromatography (TLC) used to check purity and homogeneity of synthesis compounds. FTIR spectra of the compounds in the (4000-600) cm^{-1} spectral range were recorded on SHIMAZU FTIR-8400 Fourier transform Infrared spectrophotometer using KBr discs. ^{1}HNMR and ^{13}CNMR spectra were recorded on Bruker, Ultrasheild (300) MHz in Jordan and (500) MHz in Iran,using TMS as internal reference and DMSO-d_6 as a solvent.

2.1.1 Synthesis of 2-pentadecyl-4H-benzo[1,3]oxazin-4-one [(9)] [1].

To a stirred solution of anthranilic acid (1.37gm, 0.01mole) in dry acetone, palmitoyl chloride (3ml, 0.01mole) was added in presence of pyridine as solvent. The mixture was refluxed for 3hrs then concentrated under vacuum. The solid product that separated on cold was filtrated off, dried and crystallized from petroleum-

ether. The product was heated for 4 hrs under reflux in acetic anhydride then concentrated under vacuum. Physical properties of compound [1] are listed in Table (1).

2.1.2 Synthesis of 1-(4-oxo-2-pentadecylquinazolin-3(4H)-yl) urea [2], thiourea [3], phenyl thiourea[(10)] [4]:

To a solution of compound [1] (3.57gm, 0.01mole) in 30ml pyridine,(semi carbazide, thiosemicarbazide and phenylsemicarbazide) respectively (0.01 mole) were added and the reaction mixture was heated under reflux for (6-8hrs), and poured into cold diluted HCl. the crud solid product that separated was filtered off, washed with water, dried and recrystallized from ethanol to give crystals. Physical properties of compounds [2-4] are listed in Table (1).

2.1.3 Synthesis of 3-amino-2-pentadecylquinazolin-4(3H)-one[(11)][5]:

Compound [1] (3.57gm, 0.01mole) in (10 ml) dimethylformamide (DMF) as solvent; excess of 99% hydrazine hydrate in ethanol (30ml) was added to the reaction mixture and reflexed for (6 hrs.). Finally, the reaction mixture cooled to room temperature, poured on ice-cold water, stirred and filtered. The precipitate was recrystallized from ethanol to give off white crystals. Physical properties of compound [5] are listed in Table (1).

2.1.4 Synthesis of N-(4-oxo-2-pentadecylquinazolin-3(4H)-yl) benzamide[(12)][6].

Compound [5] (3.68 gm, 0.01mole) in dry acetone (40ml) containing a catalytic amount of pyridine (3drops), (1.2ml, 0.01mole) benzoyl chloride was added in drop wise maintaining the temperature near 8° C for (1 hrs.). The reaction mixture was refluxed for another (3hrs.), and then cooled at room temperature. The separated solid product was filtered off and recrystallized from ethanol. Physical properties of compound [6] are listed in Table (2).

2.1.5 Synthesis of N-(4-oxo-2-pentadecylquinazolin-3(4H)-yl) acetamide[(12)][7].

A solution of compound [5] (3.68 gm, 0.01mole) in dry acetone (40ml) and acetyl chloride (0.8ml, 0.01mole) in pyridine (20 ml) was refluxed for (5 hrs.). The cold mixture was poured into ice/water. The separated solid was filtered off, dried and recrystallized from benzene-hexane. Physical properties of compound [7] are listed in Table (2).

2.1.6 Synthesis of N-(4-oxo-2-pentadecylquinazolin-3(4H)-yl)-2-chloro acetamide[(14)][8].

A mixture of compound [5] (3.68 gm, 0.01mole) in dry acetone and chloroacetyl chloride (0.8ml, 0.01 mole) was refluxed for 6 hrs. The mixture was then poured into cold water (30 ml) and the precipitated solid was filtered off, dried and recrystallized from benzene. Physical properties of compound [8] are listed in Table (2).

2.1.7 Synthesisof N-(4-oxo-2-pentadecylquinazoline-3(4H)-yl) carbothioamide[9], carboxamide [(15)][10].

Compound [1] (3.57gm, 0.01mole) in (15 ml) dimethylformamide (DMF) as solvent; was refluxed with (0.8gm, 0.01 mole) (thiourea and urea) respectively in (40 ml) dry acetone containing a catalytic amount of pyridine (3drops), for (10hrs.). The reaction mixture cooled to room temperature, poured on ice-cooled water, the formed precipitate was filtered and recrystallized from ethanol. Physical properties of compounds [9] and [10] are listed in Table (3).

2.1.8 Synthesis of 3-hydroxy-2-pentadecylquinazolin-4(3H)-one[(16)][11].

A solution of compound [1] (3.57gm, 0.01mole) in in (20 ml) dimethylformamide (DMF) and hydroxylamine hydrochloride (0.7gm, 0.01mole) in pyridine (25ml) was refluxed for (16hrs.). The reaction mixture was cooled and poured into a crushed ice water. The solid that separated was filtered off, washed with water, dried and then recrystallized from dioxane to give the required products. Physical properties of compound [11] are listed in Table (3).

2.1.9 Synthesis of 2-(4-oxo-2-pentadecylquinazolin-3(4H)-yl)acetic acid [(17)][12].

To a solution of compound [1] (3.57gm, 0.01mole) in (25ml) dimethylformamide (DMF), (0.75gm, 0.01mole) glycine in dry pyridine (20ml) was added and the heated under reflux for (4hrs.) then left to cool at room temperature. The reaction mixture was poured into ice/HCl to reveal the solid product, which was filtered off, washed with water, dried and recrystallized from ethanol. Physical properties of compound [12] are listed in Table (3).

2.1.10 Synthesis of 2-pentadecylquinazolin-4(3H)one[18][13].
compound [1] (3.57gm, 0.01mole) was fused with of ammonium acetate (3gm, 0.04mole) heated in an oil bath in temperature (160-170) ^{o}C for (2hrs.), then cooled, water is added, the solid product obtained after filtration and recrystallized from methanol. Physical properties of compound [13] are listed in Table (3).

2.1.11 Synthesis of 2-pentadecyl-3-(phenylamino)quinazolin-4(3H)-one[19][14].
Compound [1] (3.57gm, 0.01mole) and phenyl hydrazine (1.08gm, 0.01mole) were dissolved in absolute ethanol (50ml). This solution was refluxed for (12hrs.). Concentrated, cooled and poured into crushed ice and filtered. The solid thus obtained was purified by recrystallization from ethanol. Physical properties of compound [14] are listed in Table (3).

2.2 Conversion of the prepared compounds to nonionic surfactants [20]:
They are prepared by the addition of (n) moles of propylene oxide (n = 3, 5, 7) to one mole of synthesized compounds (2, 4, 5, 7 and 12) by mixed with KOH (0.1 wt %) The mixture was heated up to 130^{o} C and a rapid stream of N_2 gas was passed continuously for about 20 min to exclude the oxygen and water vapors. And the product was characterized by FT-IR spectrophotometer. The addition of propylene oxide gave a mixture of propenoxylated products whose structures were shown through IR spectra to be two broad bands at 1100 and 950 cm^{-1}and by ^{1}HNMR spectra showed δ= (3.2 – 3.7) ppm**.**

2.3 Determination of the performance properties

1. Surface and interfacial tensions:
Surface and interfacial tensions were measured with a Du-Nouy tensiometer (Kruss, Type 8451) using an aqueous solution of surfactants (0.1 wt %) at room temperature (25 ^{o} C) [21].

2. Cloud point:
Cloud point was determined by gradually heating a surfactant solution (1.0 wt %) in a temperature controlled bath, and recording the temperature at which the clear, or nearly clear solutions become definitely turbid. The reproducibility of this temperature was checked by cooling the solutions until they became clear again [21].

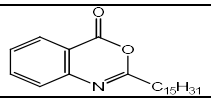
3. Wetting time:
Wetting time was determined by immersing a sample of cotton fabric in a (1.0 wt %) aqueous solution of surfactants [21].

4. Foaming properties:
Foaming properties were measured according to El-Sukkary et al (1987). In this procedure a 25 ml solution (1.0 wt %) was shaken vigorously for 10 seconds in a 100 ml graduated cylinder with glass stopper at 25 ^{o} C. The solution was allowed to stand for 30 seconds, and then, the foam height was measured [22].

5. Emulsification stability:
Emulsification stability was prepared from 10 ml of a 20 mmol aqueous solution of surfactant and 5 ml of toluene at 40 ^{o} C. Emulsion stability was determined as the time it took 9 ml of an aqueous layer to separate from the emulsion once shaking had stopped [23].

6. Determination of CMC of Surfactant by Surface Tension Method:
Surface tension measurements were done by applying du Nouy ring method using Du-Nouy (Kruss, Type 8451) tensiometer and platinum ring at room temperature (25˚C). Stock solution of 5% (v/v) of synthesized nonionic surfactant in aqua was prepared in 1 L volumetric flask. First of all, the surface tension of pure water in a glass vessel was noted. The surface tensions were measured by a dilution method in which a 5% (v/v) of synthesized nonionic surfactant stock solution was gradually diluted by deionized water. After each dilution the solution was stirred for 30 s, and then left for 2 min before reading the surface tension value. All surface tension measurements were done at least three times or up to obtaining repeated value. The CMC values were determined by plotting surface tension versus concentrations of surfactant [23].

Table (1) : Physical properties and FT-IR spectral data cm^{-1} of the synthesized compounds (1-5).

Com. No.	Physical Properties				Major FT-IR Absorption cm^{-1}				
	Structures	M.P °C	Yield %	Color	ν(C-H) arom.	ν(C-H) aliph.	ν(C=O)	ν(C=C) aromatic	Others
1		48 -50	75	Pale yellow	3030	2920 2850	1764	1598 1533 1487	ν(C=N) 1641

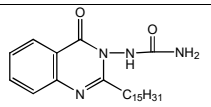
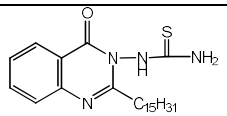
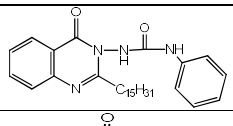
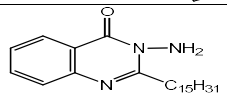
2		94-96	80	White	3061	2918 2849	1676 amid 1645 amid	1575 1531 1490	νNH 3298 νNH_2 asym 3425 sym 3226 ν(C=N) 1676
3		99-100	80	White	3062	2918 2850	1676 amid	 1585 1537 1494	ν(C=S) 1409 νNH 3358 νNH_2 asym3358 sym3286 ν(C=N)1635
4		117- 118	80	White	3052	2922 2855	1678 amid 1648 amid	1590 1538 1494	 νNH 3358 ν(C=N) 1604
5		98-100	80	Off white	3062	2916 2850	1674 amid	1590 1537 1491	νNH_2 asym3396 ym3245 ν(C=N) 1601

Table (2) : Physical properties and FT-IR spectral data cm^{-1} of the synthesized compounds (6-8).

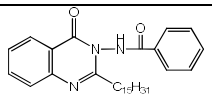
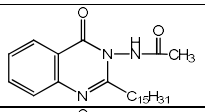
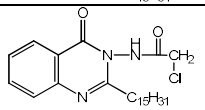
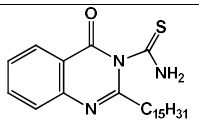
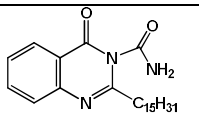
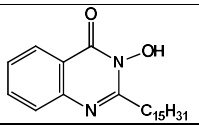
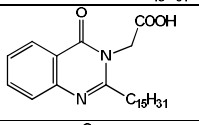
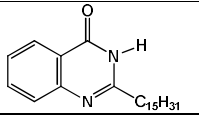
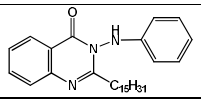
Com. No.	Physical Properties				Major FT-IR Absorption cm^{-1}				
	Structures	M.P °C	Yield %	Color	ν(C-H) arom.	ν(C-H) aliph.	ν(C=O)	ν(C=C) aromatic	Others
6		88 -90	85	White	3030	2910 2850	1689 amid	1580 1540 1492	νNH 3329 ν(C=N) 1604
7		102- 104	85	White	3035	2911 2849	1697 amid	1585 1541 1488	νNH 3329 ν(C=N) 1608
8		104- 106	85	Off white	3030	2910 2850	1685 amid 1705 amid	1585 1555 1490	νNH 3329 ν(C=N) 1635

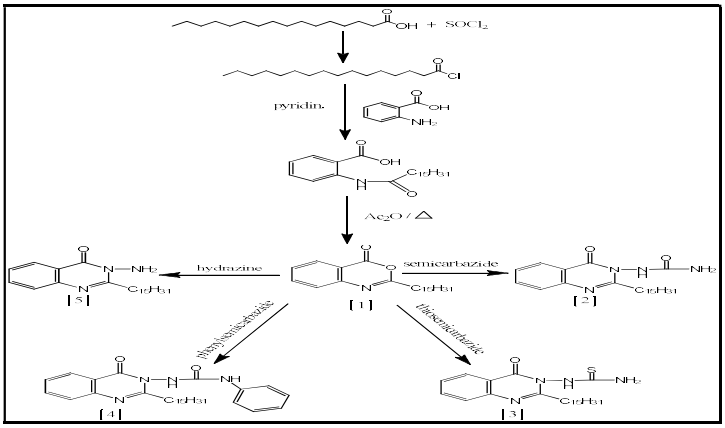
Table (3) : Physical properties and FT-IR spectral data cm^{-1} of the synthesized compounds (9-14).

Com. No.	Physical Properties				Major FT-IR Absorption cm^{-1}				
	Structures	M.P °C	Yield %	Color	ν(C-H) arom.	ν(C-H) aliph.	ν(C=O)	ν(C=C) aromatic	Others
9		90 -91	75	White	3040	2920 2860	1677 amid	 1600 1565 1494	νNH2 3440,3336 ν(C=S) 1413.7 ν(C=N) 1606.5
10		88-90	75	White	3040	2960 2860	1699 amid 1677 amid	 1585 1555 1490	νNH2 3421,3320 ν(C=N) 1608
11		72 -73	70	off White	3067	2920- 2850	1685 amid	 1605 1550 1492	νOH 3420 ν(C=N) 1604
12		92-93	75	White	3060	2945 2850	1732 acid 1697 amid	 1595 1565 1490	νOH 3340 ν(C=N) 1608
13		114 - 115	70	White	3036	2920- 2850	1681 amid	 1605 1550 1492	νNH 3168 ν(C=N) 1604

14		90-91	75	red	3050	2955 2850	1679 amid	1595 1565 1490	νNH 3340 ν(C=N) 1606

III. Results And Discussion

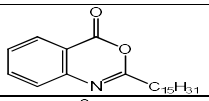
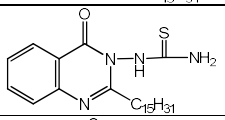
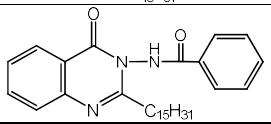
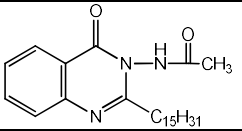
The synthetic sequences for preparation of series new 2-pentadecyl quinazolinone compounds by reaction of pentadecanoyl chloride with anthranilic acid gave 2-pentadecyl-4H-benzo[3,1]oxazin-4-one [1] which was used as starting material to synthesis some quinazolinone compounds by a reaction with nitrogen nucleophiles (e.g., semicarbazide, thiosemicrabazide, phenylsemicarbazide and hydrazine hydrate) are show in scheme(1).



Scheme (1): Synthesis of new derivatives of quinazolinone (1-5)

Physical properties of compounds [2, 3, 4 and 5] are listed in Table (1). FTIR spectrum of prepared compounds [2, 3, 4 and 5] showed appearance of absorptions at (3425-3396) cm^{-1} and at (3286-3226) cm^{-1} of νNH_2 asym. and sym. respectively. (3358, 3298) cm^{-1} of νNH group, Also it showed shift in the νC=O band from (1764cm^{-1}) of cyclic ester to (1690- 1645 cm^{-1}) of amide. FTIR spectral data are listed in Table (1). ^{1}H-NMR spectrum of compound [3] showed triplet signal at δ= (0.85) ppm due to ($-CH_3$) protons, multiplet signal at δ= (1.2 – 2.66) ppm due to (C-H) protons of long chain $14CH_2$, signals at δ= (7.11 -7.60) ppm due to aromatic rings protons, singlet signal at δ= (8.09- 8.51) ppm due to ($-NH_2$) protons, and singlet signal at δ= (11.1) ppm due to (N-H) proton as shown in Table (4). ^{13}C-NMR spectrum of compound [3] showed signals at δ= (22-27) ppm, δ= (122-133) ppm, δ= (147) ppm, δ= (162.29) ppm and δ= (174.5) ppm belong to long chain ($C_{15}H_{31}$), (C-aromatic), (C=N) imine, (C=O) ester and (C=S) respectively. ^{13}CNMR spectral data of compound [3] is listed in Table (5).

Table (4) ^{1}H-NMR spectral data (ppm) for selected compounds.

Comp. No.	Structures	^{1}HNMR Spectral data(δppm)
1		δ 0.85 (t,3H, CH_3), δ 1.59 – 2.69 (m, 28H, $14CH_2$), δ 7.1 -8.5(m, 4H,ArH)
3		δ 0.85 (t, 3H, terminal CH_3), δ 1.2 – 2.66 (m, 28H, $14CH_2$), δ 7.11 -7.60(m, 4H,ArH), δ8.09- 8.51 (s, 2H,NH_2), δ 11.1 (s,1H, NH)
6		δ 0.84 (t,3H, CH_3), δ 1.22 – 2.50 (m, 28H, $14CH_2$), δ 6.51 -7.84 (m, 9H,ArH) , δ 9.10(s, 1H ,N-NH)
7		δ 0.83 (t, 3H, terminal CH_3), δ 1.27 – 2.4 (m, 28H, $14CH_2$),δ3.47 (s,3H, O=C-CH_3), δ 7.01 -8.51(m, 4H,ArH)

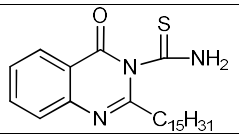
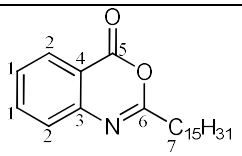
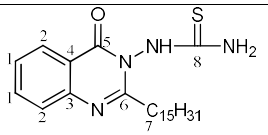
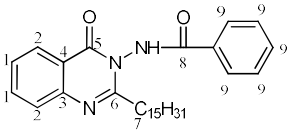
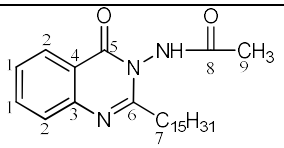
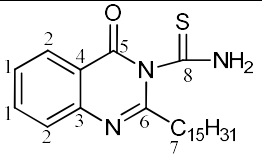
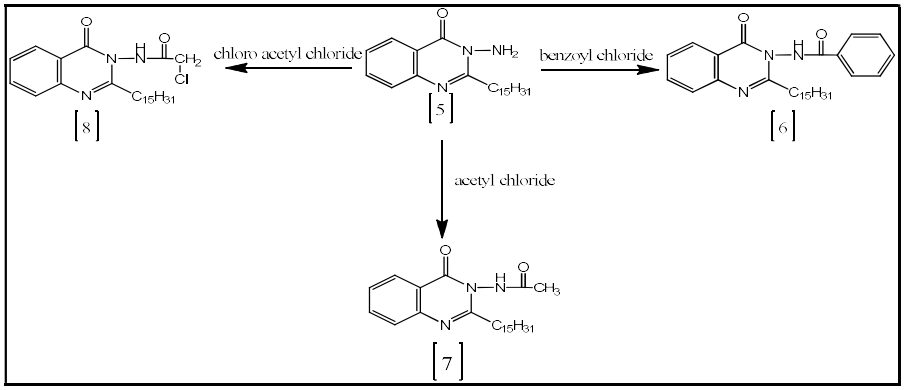
9		δ 0.85 (t,3H, CH_3), δ 1.8 – 2.38 (m, 28H, $14CH_2$), δ 7.11 -7.9(m, 4H,ArH), δ 8.5-9.14(s, 2H ,NH_2)

Table (5) : ^{13}C-NMR spectral data (ppm) for selected compounds.

Com. No	structure	13CNMR Spectral data(δppm)
1		22-27 (Long chain C_7); 122(C_1); 127(C_2)134(C_4), 147(C_3); 148(C_6); 162.4(C_5).
3		22-27 (Long chain C_7); 123(C_1); 128(C_2); 133(C_4),147(C_6); 162.92(C_5); 174.5(C_8)
6		22-27 (Long chain C_7); 122(C_1); 126(C_2); 132(C_4); 145 (C_3 and C_9) ; 146(C_6); 162 (C_5); 168 (C_8)
7		22-27 (Long chain C_7); 58 (C_9) ; 121(C_2); 128(C_4); 133.4(C_1); 145(C_3); 154(C_6); 161 (C_5); 169 (C_8)
9		17-27 (Long chain C_7); 121(C_1); 125(C_2); 134(C_4); 148(C_6); 164 (C_5);182 (C_8)

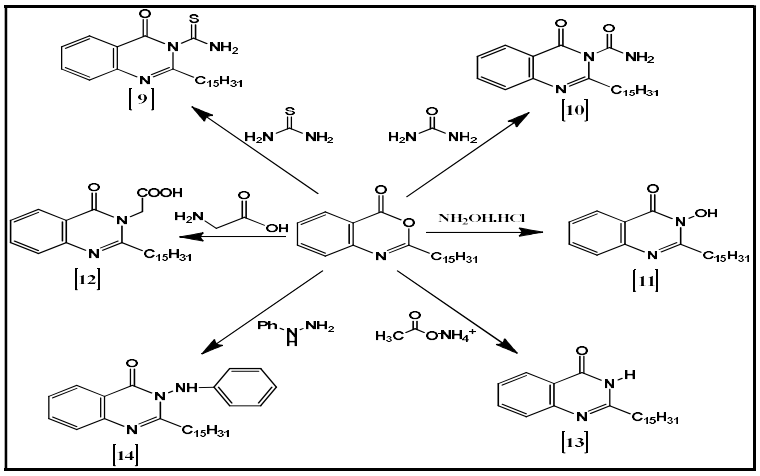
Compound [5] allowed to react with halogenated compounds such as benzoyl chloride, acetyl chloride and chloro acetyl chloride by nucleophilic substitution reaction to synthesis newly derivatives of quinazolinone compounds [6], [7] and [8]. This reaction carried out in presence of catalytic amount of pyridine and DMF. The synthetic sequences for preparation of new N-(4-oxo-2-pentadecylquinazolin-3(4H)-yl) benzamide [6], acetamide [7] and chloro acetamide [8] was performed as outlined in scheme (2).



Scheme (2)

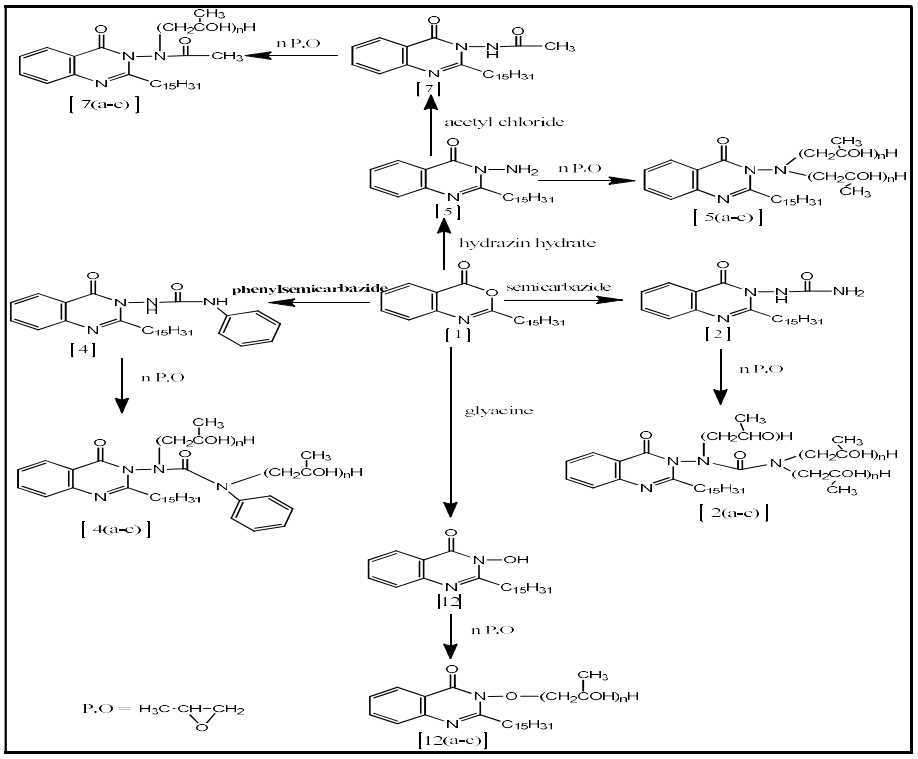
Physical properties of compounds [6], [7] and [8] listed in Table (2). FTIR spectrum of compound [6], [7] and [8] showed disappearance of ν (NH_2) absorption band at 3496 and 3245 cm^{-1} and appearance of characteristic absorption bands at ν cm^{-1} 3329 for ν (N-H), 3030 for C-H aromatic; 2910 and 2850 for C-H aliph

; 1689 for C=O (amide) ; 1604 for C=N of compound[6]; 1697 for C=O (amid) of compound[7]; and 1685for C=O (amide) of compound[8]; νC=N (1604 - 1635) cm^{-1}. ^{1}H-NMR spectrum data of compound [6] showed triplet signal at δ= (0.84) ppm due to (-CH_3) protons, multiplet signal at δ= (1.22 – 2.50) ppm due to (C-H) protons of long chain 14CH_2, signals at δ= (6.51 -7.84) ppm due to aromatic rings protons, singlet signal at δ= (9.10) ppm due to (N-H) proton. ^{1}H-NMR Spectral data of compounds [6] are listed in Table (4). ^{13}C-NMR spectrum of compound [6] showed signals at δ= (22-27) ppm, δ= (126 -135) ppm, δ= (146) ppm and δ= (168) ppm belong to long chain ($C_{15}H_{31}$), (C-aromatic), (C=N) imine and (C=O) ester respectively. ^{13}CNMR spectral data of compound [6] are listed in Table (5). While ^{1}H-NMR spectrum data of compound [7] showed triplet signal at δ= (0.83) ppm due to (-CH_3) protons, multiplet signal at δ= (1.27 – 2.4) ppm due to (C-H) protons of long chain 14CH_2, singlet signal at δ= (3.47) ppm due to (O=C-CH_3) proton, signals at δ= (7.01 -8.51) ppm due to aromatic rings protons. ^{1}H-NMR Spectral data of compounds [7] are listed in Table (4). ^{13}C-NMR spectrum of compound [7] showed signals at δ= (22-27) ppm, δ= 58 ppm, δ= (128-134) ppm, δ= (145) ppm, δ= (154) ppm, δ= (161) ppm and δ= (169) ppm belong to long chain ($\underline{C}_{15}H_{31}$), (O=$\underline{C}$-CH_3), (C- aromatic), ($\underline{C}$=N) imine , ($\underline{C}$=O) ester ($\underline{C}$=O-CH_3) respectively. ^{13}CNMR spectral data of compound [7] are listed in Table (5). The syntheses of different new quinazolinone compounds by reaction of compound [1] with different substituted amine compounds. These different synthesized compounds that are presented in Scheme (3).



Scheme (3)

Reaction of compound [1] with thiourea and urea in presence catalytic amount of pyridine afforded the product N-(4-oxo-2-pentadecylquinazoline-3(4H)-yl) carbothioamide [9], carboxamide [10] respectively. Physical properties of compounds [9] and [10] are listed in Table (3). FTIR spectrum of prepared compounds [9-10] showed appearance of absorptions at (3440-3320) cm^{-1} , (3040) cm^{-1} , (2960, 2860) cm^{-1} , (1699 and 1677) cm^{-1}, (1608) cm^{-1} due to ν –NH_2, ν C-H aromatic, ν C-H aliphatic, ν C=O for amide, ν C=N of compounds (9 and 10), in addition (1413) cm^{-1} for ν C=S of compound [9]. In the other hand^{1}HNMR spectra data of compound (9) δ ppm in DMSO-d_6 solvent showed singlet signal at δ= (0.85) ppm due to (-CH_3) protons, multiplate signale at δ= (1.18-2.38) due to long chain (14CH_2), signals at δ= (7.9-7.11) ppm due to aromatic ring protons and δ= (8.5-9.14) ppm due to (-NH_2) protons. As listed in table (4). ^{13}C-NMR spectrum data of compound [9] showed signals at δ= (17-27) ppm, δ= (121-134) ppm, δ= (148) ppm, δ= (164.07) ppm and δ= (182.3) ppm belong to long chain (-$C_{15}H_{31}$), (C-aromatic), (C=N), (C=O) amide and (C=S) respectively.^{13}CNMR spectral data of compound [9] is listed in Table (5).. the synthesized quinazolinone derivatives bearing an active hydrogen atom such as (-NH, -OH, -COOH and - NH_2) and the structure of a surface active agent requires a hydrophilic component. This is accomplished through the condensation of propylene oxide with different moles, (3, 5 and 7) moles in the presence of KOH as catalyst at any active hydrogen atom of synthesized compounds [2, 4, 5, 7 and 12] which contain one or more active hydrogen atoms to yield novel groups of nonionic surfactant (2(a-c), 4(a-c), 5(a-c), 7(a-c) and 12(a-c)) respectively that showed in scheme (4).



Scheme (4)

The study of the surface active properties of the polypropenoxy compounds has been done in an aqueous solution (1wt %, pH = 7) at 25°C. The surface activity and related properties of the synthesized compounds including surface tension and interfacial tension, wetting time, cloud point, foaming height, emulsification properties and CMC are given in Table (6).

The surface and interfacial tension of the synthesized compounds are shown in Table (6). It can be observed that the nonionic surfactants have pronounced surface activity. The surface and interfacial tension increase with an increase in the molecular weight of the hydrophobic moiety. The data given in Table (6) shows that the values of surface and interfacial tension increased with the increase in the number of propylene oxide units added to the molecule. (24)

A very important factor in making the most efficient use of nonionic surfactants in an aqueous system is an understanding of the property called cloud point. All synthesized compounds showed high cloud points which gave performance in hot water and it was increased by increasing the number of the propoxy group.

All the synthesized compounds showed a decrease in wetting time with an increase in the number of propylene oxide units in the molecule. Moreover, the presence of propylene oxide in different moles caused a reduction in wetting time, i.e. improving their wetting properties which make widely applicable in the textile industry. (25)

Nonionic surfactants containing an aromatic ring such showed poor foaming properties. The foam height of the prepared surfactants increases with an increase in the number of propylene oxide units per molecule of surfactant. The low foaming power could have an application in the dyeing auxiliary industry

Emulsification is one of the most important Properties of surfactants. In many textile processes such as scouring and dyeing, it is necessary to introduce surfactants into the bath to remove oily impurities from the fibers. On the other hand, nonionic surfactants with good emulsion stability have been used in such fields as, shampoos, cosmetics, emulsion paints and the textile industry. The results in Table (6) showed that the emulsion stability increases by decreasing the number of propylene oxide units.

Table (6) surface properties of synthesized compounds (2(a-c), 4(a-c), 5(a-c), 7(a-c) and 12(a-c))

compd	No of moles	Surface Tension (dyne/cm) 0.1wt%	Interfacial tension (dyne/cm) 0.1wt%	Cloud point C 0.1wt%	Wetting time (Sec) 0.1wt%	Emulsion stability (Min) 25 mmole	Foam height (mm) 0.1wt%	CMC × 10^{-3}
2(a-c)	3 5 7	32 33 35	10 11 13	58 64 73	43 32 21	92 85 74	85 115 135	3.6 4.1 4.3
4(a-c)	3 5 7	30 33 34	9 11 12	67 75 84	46 28 20	94 84 71	90 105 130	3.8 4.0 4.2
5(a-c)	3 5 7	28 30 32	8 10 12	74 91 98	41 33 24	101 95 75	95 110 140	3.9 4.1 4.3
7(a-c)	3 5 7	32 33 35	9 11 13	68 82 91	49 35 26	96 85 78	105 135 150	4.0 4.1 4.4
12(a-c)	3 5 7	33 34 35	10 13 14	72 76 81	40 31 21	112 93 87	95 120 135	3.4 3.6 3.9

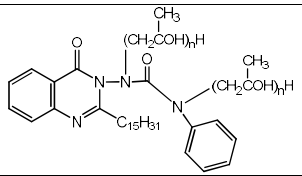
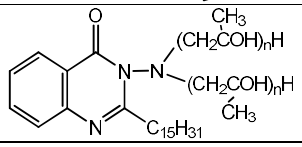
The reaction condition of propenoxilated compounds are illustrated in table (7).

Table (7) reaction condition of propenoxilated compounds

compd	Catalyst,wt%	Temp C	Propenoxylated compounds	Yield%	Degree of propenoxylation	color
2	KOH, 0.01wt%	130	2(a-c)	55-60	3,5 and 7	brown cream
4			4(a-c)	60-65		Pale brown cream
5			5(a-c)	70-74		Yellow white Cream
7			7(a-c)	63-68		Pale brown cream
12			12(a-c)	72-75		Yellow white Cream

^{1}H-NMR spectra data of compound [4(a-c) and 5(a-c)] showed disappearance peaks –NH of compound [4] and $–NH_2$ of compound [5]. And appearance multiplate signal at δ= (3.2 – 3.7) ppm due to the protons of propenoxy groups ($-CH_2-CH-(CH_3)-O-$)- beside other characteristic bands as listed in table (8) and shown in Figure (1 and 2).

Table (8): ^{1}H-NMR spectral data δ ppm for compound [4(a-c) and 5(a-c)]

Comp. No.	Structures	^{1}HNMR Spectral data(δppm)
4(a-c)		δ 0.83 (t,3H, CH_3), δ 1.8 – 2.38 (m, 28H, $14CH_2$), δ 3.2-3.7(m, ($-CH_2-CH-(CH_3)-O-$) δ 7.11 -7.9(m, 9H,ArH)
5(a-c)		δ 0.85 (t,3H, CH_3), δ 1.8 – 2.38 (m, 28H, $14CH_2$), δ 3.2-3.7(m, ($-CH_2-CH-(CH_3)-O-$) δ 7.11 -8.3(m, 4H,ArH)

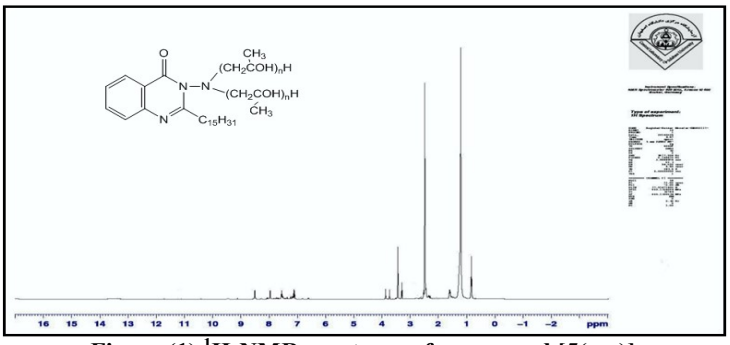


Figure (1) ^{1}H-NMR spectrum of compound [5(a-c)]

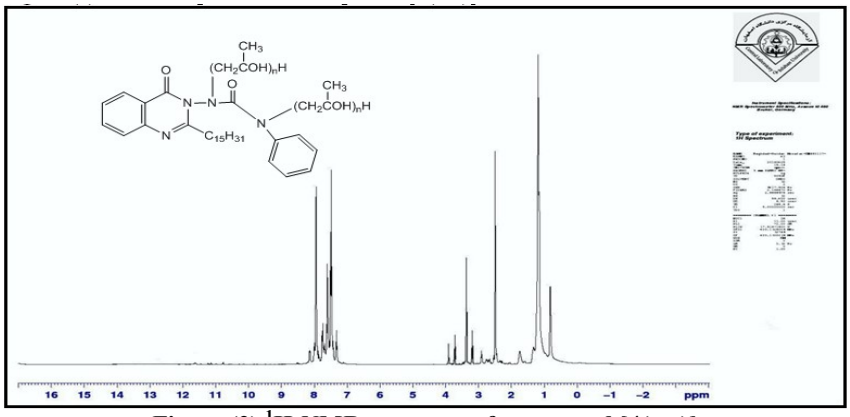


Figure (2) ^{1}H-NMR spectrum of compound [4(a-c)]

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