

MINIMIZING EVAPORATION OF LIGHT HYDROCARBONS FOR IRAQI GASOLINE BY USING D-GLUCITOL FATTY ACID ESTERS AS REDUCED PRESSURE AGENTS, SYNTHESIS AND CHARACTERIZATION

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## Abstract

The study involved synthesis of [1,6-Di-O-(4-Alkanoyloxybenzoyl)-D-glucitol by reaction of 4-alkanoyloxy benzoyl chloride with D-glucitol in 2:1 molar ratio in pyridine as a solvent, all the reactions monitored by TLC and the structures were characterized using FTIR technique. Synthesized compounds possibility of reducing pressure were estimated by lowering vapor pressure for concentrations (1-10 mg L<sup>-1</sup>) at various temperatures (37.8, 40, 43 and 45°C). Saturated vapor pressure (Reid vapor pressure) and surface tension showed successful attempt for using synthesized D-Glucitol fatty acid esters as agents for reducing gasoline pressure at various concentrations and temperatures. These compounds were reduced the surface tension as average 3 mN/m for Benzoic acid and D-glucitol fatty acid ester compounds, while reduced vapor pressure as average 18.8 kPa for Benzoic acid fatty acid ester and 25 kPa for D-glucitol fatty acid ester compounds respectively.

**Keywords:** *D-glucitol fatty acid ester; Benzoic acid fatty acid ester; gasoline pressure; Reid vapor pressure.*

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## 1. Introduction

Accumulating the nonpolar segment, commonly called "the tail" and the other polar segment called "the head" in same molecule provides requirements of amphiphilic molecules (Surfactant) such as sugar fatty acid esters (SFAEs). These compounds can be produced by using either chemically [1] or enzymatically [2-8]. These compounds featured nonionic surfactants and used in foods, pharmaceutical and cosmetics because they are odorless, tasteless, biodegradable, nontoxic and nonirritant [9-11]. Some of sugar fatty acid esters (SFAEs) are insecticidal and miticidal [12]. Physical properties of them were varying by sugar group and number of carbon atoms in alkyl chain [13]. Surfactant have wide variety applications involving wetting, dispersing, foaming, detergency and emulsification agents [14]. Due to the different segments for surface active compounds, they are lined up to form aggregates called micelles at concentration called critical micelle concentration (CMC) which may be formed in either polar or non-polar solvents. In the nonpolar solvent, reversed or inverted micelles formed, and with the same nonpolar solvent, the hydrophilic segment shielded from solvent by hydrophobic segment, while in polar solvent normal micelles formed, and the hydrophobic segment shielded from solvent by hydrophilic segment [15] (see Figure 1).

The properties of surfactant agents such as surface tension and conductivity were changed with the length of hydrophobic tail and concentration of CMC [16-18]. All countries suffering from losing of gasoline and light hydrocarbons via evaporating which caused environmentally and economic problems. For example, In Russia 500-600 thousands tons of oil were lost by evaporation yearly [19-21]. It is known that the temperature of weather in Iraq up to 45°C during the summer. This makes fuel station owners in Iraq sufferers from evaporation of gasoline during storage, refueling and transport then increasing polluter in the environment, so refinery most recompense fuel station owners by additional quantity of gasoline rather than

evaporated, this causes of the economic problem. The purpose of this paper is synthesis of 4-hydroxy benzoic acid and -D-glucitol fatty acid esters derivatives, as reducing pressure agents, therefore the reduction of gasoline vapor pressure then reduces evaporation for a healthy environment and reduces economic losses.

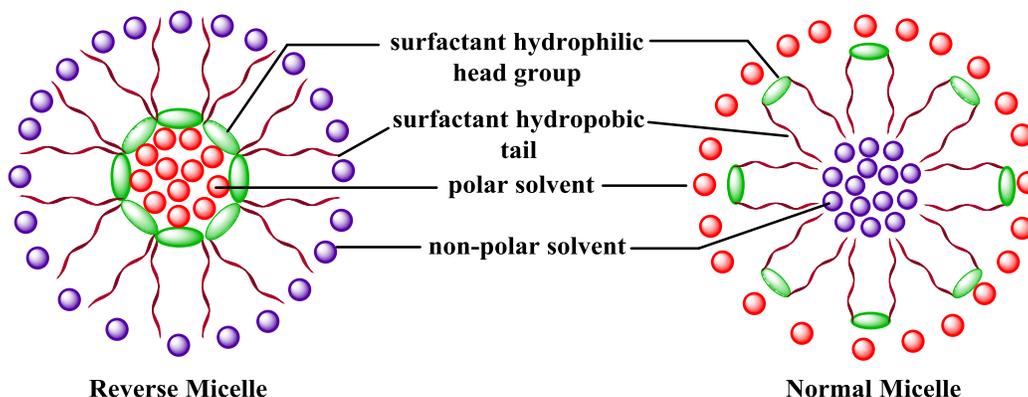


Figure 1. Normal and reverse Micelle

## 2. Materials and methods

### 2.1. Materials

D-glucitol, 4-hydroxy benzoic acid, myristic acid, palmitic acid, stearic acid and thionyl chloride Sigma-Aldrich Chemical Co. were used without further purification, Solvents Scharlau Chemical Co. Surface tension was performed at 25°C by capillary tube method, melting point was measured on JENWAY apparatus, FTIR spectra were recorded as Attenuated total reflection (ATR) on Bruker-Tensor 27 Spectrometer, UV-Vis spectra were record by UV/visible Jenway spectrophotometer Model 6800, solvent THF.

### 2.2. Methods

#### 2.2.1. Procedure for synthesis

##### i. Alkanoyloxy benzoic acid ( $A_{14}$ , $A_{16}$ and $A_{18}$ )

To two necks 100 mL RB flask contains 30 mL dry THF and (14.5 mmol) of 4-hydroxy benzoic acid and few drops of  $Et_3N$  was added (14.5 mmol) of appropriate acid chloride mixed with 10 mL dry THF through dropping funnel at 0°C under  $N_2$  gas flow, the mixture was stirred at RT overnight, then poured on crush ice with stirring after 30 min. Filtered, washed with distilled water (50 mL), dried and recrystallized from dichloromethane.

##### ii. 4-Alkanoyloxy benzoyl chloride ( $B_{14}$ , $B_{16}$ and $A_{18}$ )

In 100 mL RB flask contains 20 mL dry THF and (5 mmol) of 4-alkanoyloxy benzoic acid ( $A_{14}$ ,  $A_{16}$  and  $A_{18}$ ) was added (10 mmol) of thionyl chloride, the mixture refluxed for 2 hr. in water bath at 75 °C, then distilled under reduce pressure to remove the solvent and excess of thionyl chloride to give the desired acid chlorides.

##### iii. 1,6-Di-O-(4-Alkanoyloxybenzoyl)-D-Glucitol ( $C_{14}$ , $C_{16}$ and $C_{18}$ )

To two necks 100 mL RB flask immersed in ice contains 50 mL dry pyridine and (3 mmol) of D-glucitol was added drop by drop (6 mmol) of appropriate 4-Alkanoyloxy Benzoyl chloride ( $B_{14}$ ,  $B_{16}$  and  $B_{18}$ ) through dropping funnel under  $N_2$  gas flow, the mixture was stirred at RT overnight, then acidified with 10 ml of 25% HCl then poured on crush ice with stirring, obtained product was filtered, washed with 5% HCl then successive with distilled water.

#### 2.2.2. Characterization of 4-Alkanoyloxy Benzoic Acid ( $A_{14}$ , $A_{16}$ and $A_{18}$ )

FTIR, UV/visible and some data of physical properties for (4-Alkanoyloxy benzoic acid) are listed below.

**i. Myristyloxy Benzoic Acid [A<sub>14</sub>]**

White solid, yield 87%; mp 97 °C. IR (ATR,  $\nu$  cm<sup>-1</sup>): 2500-3446 (O-H Carboxylic acid), 3026 (C-H aromatic), 2917, 2844 (C-H aliphatic), 1694 (C=O), 1581, 1514 (C=C aromatic), 721 (bending C-H aliphatic). UV/visible (THF) nm: 253  $\pi \rightarrow \pi^*$ (rings), TLC:  $R_f$  =0.61 (toluene: methanol)(8:2).

**ii. Palmitoyloxy Benzoic Acid [A<sub>16</sub>]**

White solid, yield 85%; mp 58 °C. IR (ATR,  $\nu$  cm<sup>-1</sup>): 2500-3400 (O-H Carboxylic acid), 3028 (C-H aromatic), 2913, 2847 (C-H aliphatic), 1694 (C=O), 1608, 1503 (C=C aromatic), 717 (bending C-H aliphatic). UV/visible (THF) nm: 251  $\pi \rightarrow \pi^*$ (rings), TLC:  $R_f$  =0.59 (toluene: methanol)(8:2).

**iii. Stearoyloxy Benzoic Acid [A<sub>18</sub>]**

White solid, yield 81%; mp 55 °C. IR (ATR,  $\nu$  cm<sup>-1</sup>): 2500-3400 (O-H Carboxylic acid), 3026 (C-H aromatic), 2914, 2847 (C-H aliphatic), 1697 (C=O), 1569, 1499 (C=C aromatic), 723 (bending C-H aliphatic). UV/visible (THF) nm: 253  $\pi \rightarrow \pi^*$ (rings),. TLC:  $R_f$  =0.60 (toluene: methanol) (8:2).

**2.2.3. FTIR data for (4-Alkanoyloxy benzoyl chloride) are listed below****i. Myristyloxy Benzoyl Chloride [B<sub>14</sub>]**

Yellow liquid, yield 90%, d. 0.879 gm/mL. IR (ATR,  $\nu$  cm<sup>-1</sup>): 3048 (C-H aromatic), 2925 (C-H aliphatic), 1797 (C=O), 1602, 1515 (C=C aromatic), 722 (bending C-H aliphatic).

**ii. Palmitoyloxy Benzoyl Chloride [B<sub>16</sub>]**

Yellow liquid, yield 90%, d. 0.889 gm/mL. IR (ATR,  $\nu$  cm<sup>-1</sup>): 3047 (C-H aromatic), 2922 (C-H aliphatic), 1797 (C=O), 1601, 1510 (C=C aromatic), 721 (bending C-H aliphatic).

**iii. Stearoyloxy Benzoyl Chloride [B<sub>18</sub>]**

Yellow liquid, yield 87%, d. 0.966 gm/mL. IR (ATR,  $\nu$  cm<sup>-1</sup>): 3047 (C-H aromatic), 2922 (C-H aliphatic), 1799 (C=O), 1601, 1511 (C=C aromatic), 721 (bending C-H aliphatic).

**2.2.4. Characterization of (1,6-Di-O-(4-Alkanoyloxy benzoyl)-D- Glucitol) (C<sub>14</sub>, C<sub>16</sub> and C<sub>18</sub>)**

The following characterization data for D-Glucitol fatty acid esters.

**i. (1,6-Di-O-(4-Myristyloxy benzoyl)-D- Glucitol), [C<sub>14</sub>]**

White gum, 71%. IR (ATR,  $\nu$  cm<sup>-1</sup>): 3329 (O-H), 3028 (C-H aromatic), 2920 (C-H aliphatic), 1731 (C=O), 1606, 1499 (C=C aromatic), 721 (bending C-H aliphatic). UV/visible (THF) nm: 251  $\pi \rightarrow \pi^*$ (rings). TLC:  $R_f$  =0.87 (Benzene: methanol)(9:1).

**ii. (1,6-Di-O-(4-Palmitoyloxy benzoyl)-D- Glucitol), [C<sub>16</sub>]**

White gum, 76%. IR (ATR,  $\nu$  cm<sup>-1</sup>): 3325 (O-H), 3029 (C-H aromatic), 2916 (C-H aliphatic), 1734 (C=O), 1616, 1528 (C=C aromatic), 722 (bending C-H aliphatic). UV/visible (THF) nm: 247  $\pi \rightarrow \pi^*$ (rings). TLC:  $R_f$  =0.87 (Benzene: methanol)(9:1).

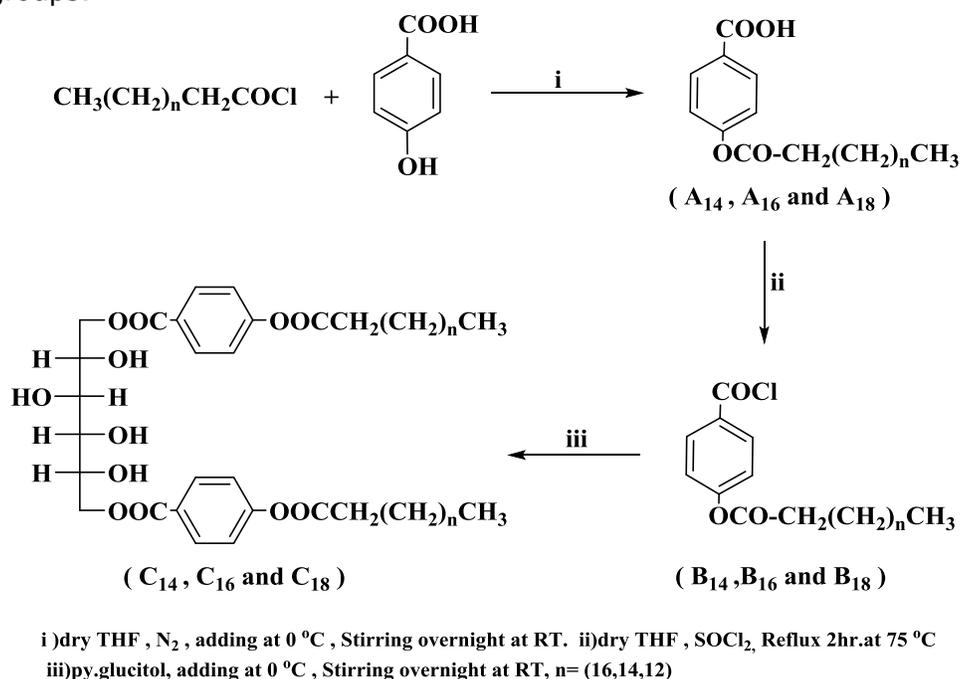
**iii. (1,6-Di-O-(4-Stearoyloxy benzoyl)-D- Glucitol), [C<sub>18</sub>]**

White gum, 69%. IR (ATR,  $\nu$  cm<sup>-1</sup>): 3346 (O-H) , 3026 (C-H aromatic), 2914 (C-H aliphatic ), 1735 (C=O), 1616, 1528 (C=C aromatic), 722 (bending C-H aliphatic ). UV/visible (THF) nm: 252  $\pi \rightarrow \pi^*$ (rings). TLC:  $R_f$  =0.87 (Benzene: methanol)(9:1).

**3. Result and discussion**

The desired compounds were synthesized according to the route illustrated in scheme 1. The scheme shows initial exothermic esterification reactions for phenolic hydroxyl; therefore, the reactions were carried out in an ice bath, followed by conversion of acids to their chlorides by reaction of acids with thionyl chloride. Finally, esterification reaction of group sorbitol was carried out by using acid chlorides and pyridine as a solvent, all esterification reactions of sorbitol were taking place in ice bath due to absorbing of emitted heat and prevent reactions on inner hydroxyl groups. The repulsion between non-bonding electrons on oxygen atoms for hydroxyl groups with electronic cloud of n orbitals for aromatic rings assist to orient esterifi-

cation reactions towards only positions 1 and 6, due to effect of repulsion, which occur between two hydroxyl groups, while esterification reaction using inner (2,3,4 and 5 hydroxyl groups) gave the repulsion which will occurs between four hydroxyl groups in addition steric bulky for groups.



Scheme 1. Synthetic route of surfactant compounds

### 3.1. Surface tension measurements

Surface tension measurements consider as important way for studying aggregation behavior of nonionic surfactants [22]. Surface tension for six synthesized surfactants was evaluated for concentrations (1 - 10 mg L<sup>-1</sup>) at 25 °C, results are listed in (table 1).

Table 1. Surface tension mN/m for synthesized compounds.

mg L <sup>-1</sup>	A <sub>14</sub>	A <sub>16</sub>	A <sub>18</sub>	C <sub>14</sub>	C <sub>16</sub>	C <sub>18</sub>
0	22.1637	22.1637	22.1637	22.1637	22.1637	22.1637
1	20.2294	20.5015	19.9641	20.4101	20.6038	20.6101
2	19.8708	19.9242	19.8655	20.8060	20.7369	19.7026
3	19.8908	19.9027	19.6682	20.0250	19.9740	19.9990
4	19.8681	20.1116	19.6664	18.5892	19.4010	18.9892
5	19.6812	20.2918	19.6297	19.7090	20.0871	19.7090
6	18.7827	20.0533	19.5987	19.4741	20.0512	19.7741
7	19.1843	19.3393	18.9991	20.1664	20.5899	20.3664
8	19.3553	20.2490	19.7076	20.2140	20.5690	20.1140
9	19.9396	20.2420	19.6664	19.8567	20.2391	20.1567
10	19.7615	20.2917	19.4629	20.0249	20.6165	20.0249

The values in Table 1, indicate that all synthesized compounds having surface active properties due to their ability to decrease surface tension. Moreover the dependence of Surface tension on surfactant concentration (Figure 2 and 3)

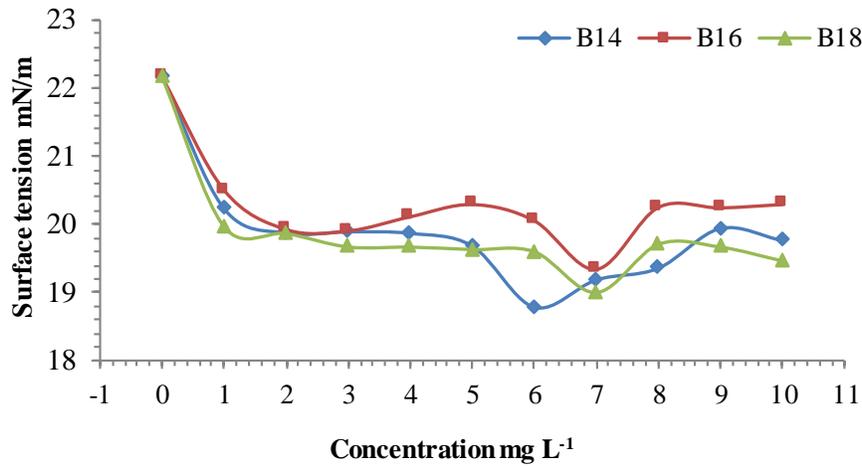


Figure 2. Dependence of surface tension on the surfactant concentration (B<sub>14</sub>, B<sub>16</sub> and B<sub>18</sub>)

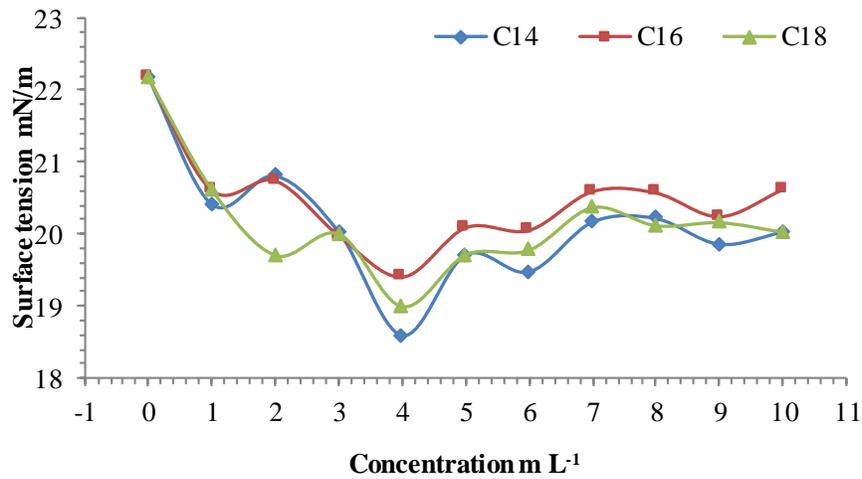


Figure 3. Dependence of surface tension on the surfactant concentration (C<sub>14</sub>, C<sub>16</sub> and C<sub>18</sub>)

The graphs in (Figure 2 and Figure 3) indicate that concentrations (6 and 7 mg L<sup>-1</sup> for B<sub>x</sub> compounds) and (4 mg L<sup>-1</sup> for C<sub>x</sub> compounds) respectively most active concentration to reduce surface tension.

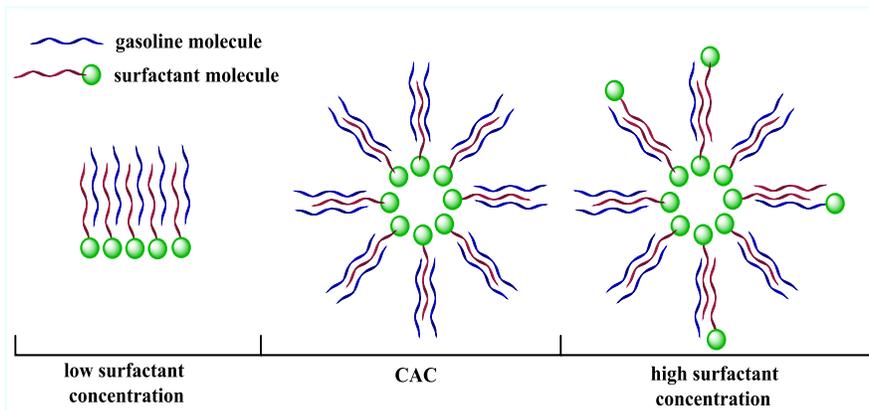


Figure 4. Binding of gasoline molecule with surfactant at different concentration

This concentration called critical aggregation concentration CAC, subsequently addition concentration leads to rising of surface tension values, this behavior can be explained by (Figure 4), before CAC stage, surfactant concentration insufficient to form aggregation to binding with gasoline molecule, at CAC be available enough by binding sites for gasoline molecule with formed aggregations, at concentration more than CAC hydrophobic tail for addend surfactant compete gasoline molecule to binding with formed aggregation, then decreasing aggregation ability to attract gasoline molecule on surface –weather interface.

### 3.2. Saturated vapor pressure

Saturated vapor pressure (Reid Vapor Pressure RVP) was evaluated by preparation of ten concentrations (1 - 10 mg L<sup>-1</sup>) for every synthesized compound at (37.8, 40, 43 and 45°C) as simulation to different temperature of air in Iraq and compared with free gasoline.

#### 3.2.1. Saturated vapor pressure for ( B<sub>14</sub>, B<sub>16</sub> and B<sub>18</sub> ) compounds

Table 2 and Figures (5, 6 and 7) show results of saturated vapor pressure (RVP) for surfactants (4-alkanoyloxy benzoic acid) were investigated.

Table 2. Saturated Vapor Pressure (kPa) for B<sub>14</sub>, B<sub>16</sub> and B<sub>18</sub> compounds

		Concentration mg L <sup>-1</sup>											
		0	1	2	3	4	5	6	7	8	9	10	°C
Comp.	B <sub>14</sub>	59.1	58.5	58.6	55.4	55.3	53.1	56.9	54.9	53.3	49.9	54.0	37.8
		62.3	63.8	61.8	55.7	53.9	53.8	57.8	57.4	52.9	52.0	55.6	40
		67.7	68.1	66.2	61.9	56.4	57.1	58.6	55.1	55.4	52.3	57.3	43
		70.5	69.2	67.0	60.5	60.1	56.1	58.5	57.0	57.8	54.4	59.2	45
Comp.	B <sub>16</sub>	59.1	56.8	54.8	51.9	53.2	52.2	53.8	55.9	54.8	50.2	55.7	37.8
		62.3	59.1	56.5	52.7	52.2	51.6	52.9	51.8	51.4	51.1	57.1	40
		67.7	55.9	54.8	54.2	55.2	55.3	55.1	54.9	52.8	51.8	58.5	43
		70.5	64.9	63.5	57.1	54.7	55.5	55.2	55.9	55.1	52.9	58.9	45
Comp.	B <sub>18</sub>	59.1	56.1	41.1	39.8	44.9	40.5	35.4	36.3	33.9	30.1	38.3	37.8
		62.3	61.7	54.5	44.6	44.3	46.5	47.8	46.9	41.1	33.9	46.9	40
		67.7	49.7	45.1	50.1	49.9	44.2	50.0	50.9	51.0	35.6	45.1	43
		70.5	68.8	64.8	46.9	53.2	50.2	53.8	55.9	56.0	38.9	48.6	45

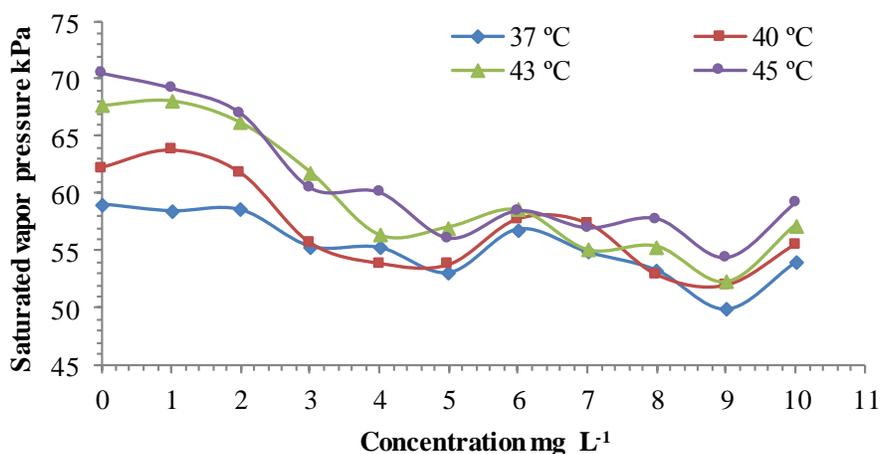


Figure 5. Dependence of saturated vapor pressure on B<sub>14</sub> concentration

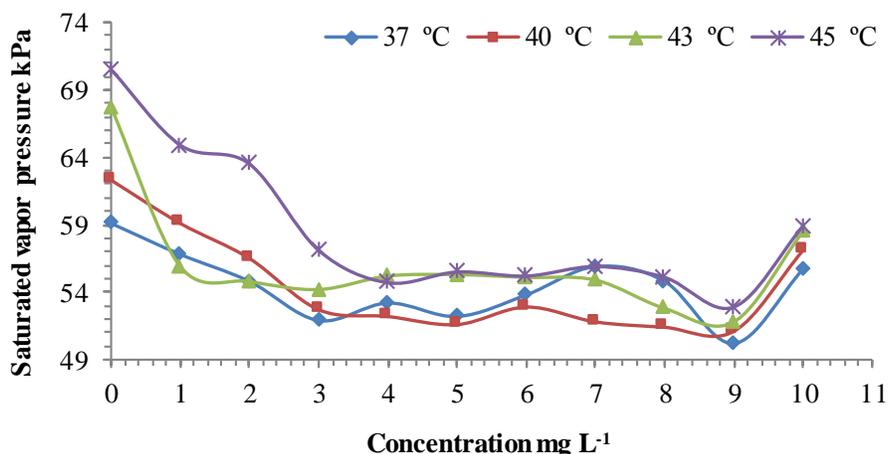


Figure 6. Dependence of saturated vapor pressure on B<sub>16</sub> concentration

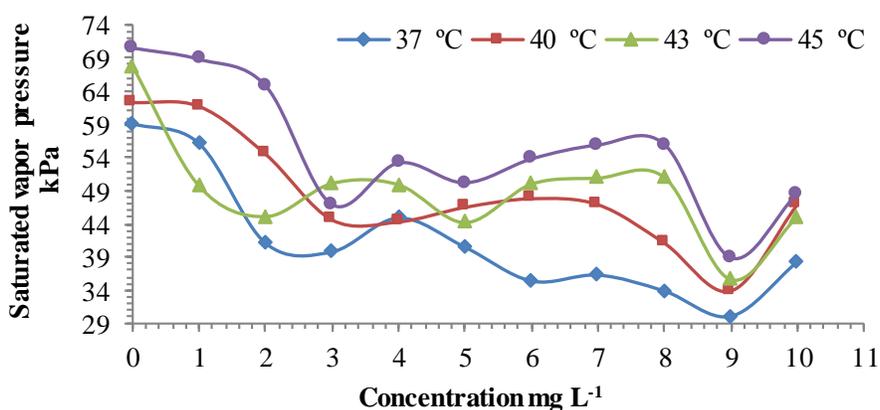


Figure 7. Dependence of saturated vapor pressure on B<sub>18</sub> concentration

Results of saturated vapor pressure of compounds ( B<sub>14</sub> , B<sub>16</sub> and B<sub>18</sub> ) show that addend 9 mg L<sup>-1</sup> most active concentration due to minimize vapor pressure in comparison with free gasoline, this behavior can be explained by mole fraction  $x_i$  and  $P_i$  saturated vapor pressure of the hydrocarbon on gasoline surface, when surface layer contains concentration of surfactant, the concentration of gasoline reduced therein, then the mole fraction  $x_i$  of gasoline reduced, in accordance with vapor pressure  $P = \sum P_i x_i$ , reduces the saturated vapor pressure according to Raoult-Dalton's law.

### 3.2.2. Saturated vapor pressure for (C<sub>14</sub>, C<sub>16</sub> and C<sub>18</sub>) compounds

At different concentrations (1 – 10 mg L<sup>-1</sup>) and temperatures (37.8, 40, 43 and 45°C) saturated vapor pressure (RVP) for (1,6-Di-O-(4-alkanoyloxy benzoyl)-D-Glucitol) as surfactant was investigated, results are listed in table 3 and graphs (Figure 8, Figure 9 and Figure 10).

Saturated vapor pressure results for D-glucitol fatty acid esters (C<sub>14</sub>, C<sub>16</sub> and C<sub>18</sub>) shows that addend 4 mg L<sup>-1</sup> of (C<sub>16</sub> and C<sub>18</sub>) and 7 mg L<sup>-1</sup> of (C<sub>14</sub>) minimize vapor pressure, and when comparing this results with active concentration for ( B<sub>14</sub>, B<sub>16</sub> and B<sub>18</sub> ) compounds, its found that required quantity of C<sub>x</sub> less than B<sub>x</sub> compounds, this is due to the large size of -D-glucitol fatty acid esters molecule, which reduces the number of molecules needed to form the aggregations required to lowering the pressure and it contains two hydrocarbon chains in addition to its ability to form four hydrogen bonding between two molecules.

Table 3. Saturated Vapor Pressure (kPa) for C<sub>14</sub>, C<sub>16</sub> and C<sub>18</sub> compounds

		Concentration mg L <sup>-1</sup>											
		0	1	2	3	4	5	6	7	8	9	10	°C
Comp.	C <sub>14</sub>	59.1	69.9	67.3	51.2	49.6	46.1	48.2	44.0	45.6	51.2	50.3	37.8
		62.3	75.5	70.0	49.2	56.1	48.7	48.1	38.9	42.9	55.9	50.1	40
		67.7	78.0	66.9	55.0	52.1	51.1	60.1	48.3	53.9	62.4	54.1	43
		70.5	72.3	66.9	60.6	61.9	52.9	61.8	51.3	54.2	53.4	51.9	45
C <sub>16</sub>	59.1	59.5	54.4	38.1	34.1	35.2	34.9	39.7	42.3	37.9	36.8	37.8	
	62.3	59.1	45.2	46.5	37.7	39.9	38.7	49.1	53.2	50.9	50.0	40	
	67.7	53.3	43.1	49.2	39.4	45.2	48.1	45.9	51.1	48.7	47.1	43	
	70.5	62.4	59.3	52.8	40.5	53.7	53.1	57.1	55.1	54.8	51.2	45	
C <sub>18</sub>	59.1	50.1	42.5	42.6	36.2	40.1	41.0	40.5	42.2	40.6	40.6	37.8	
	62.3	45.8	48.1	38.9	33.7	37.9	42.6	40.2	36.1	44.6	42.1	40	
	67.7	63.9	44.9	45.9	37.1	43.8	42.7	42.3	41.8	40.7	45.7	43	
	70.5	53.2	51.8	48.1	38.1	48.1	45.1	45.3	46.9	44.1	52.8	45	

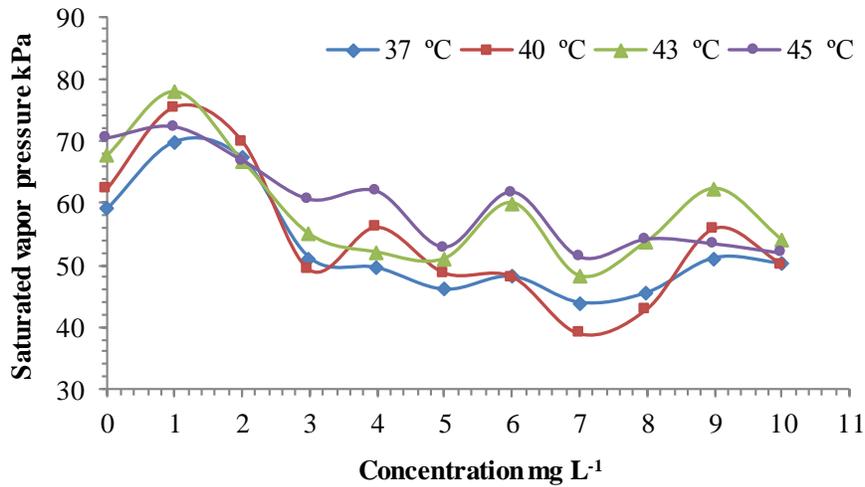


Figure 8. Dependence of saturated vapor pressure on C<sub>14</sub> concentration

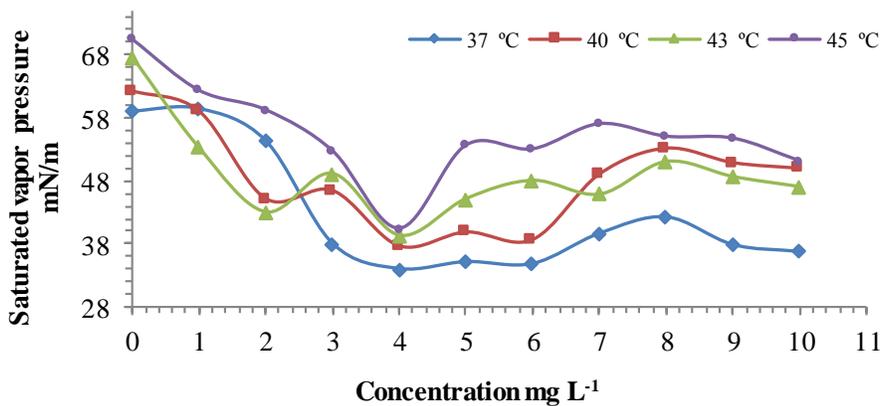


Figure 9. Dependence of saturated vapor pressure on C<sub>16</sub> concentration

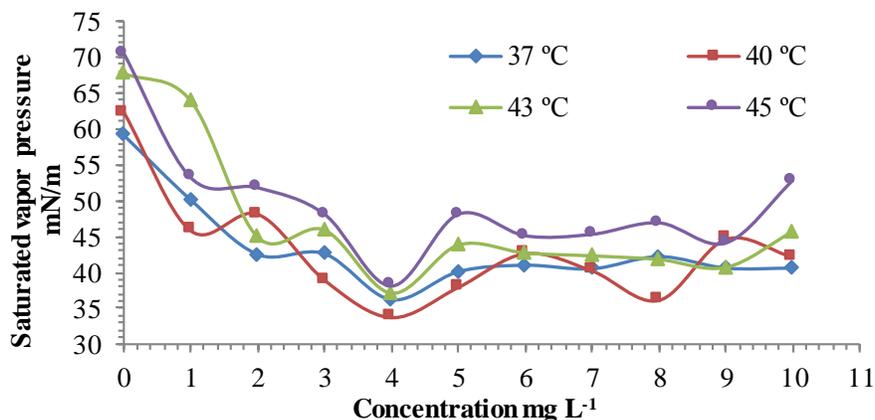


Figure 10. Dependence of saturated vapor pressure on C<sub>18</sub> concentration

#### 4. Conclusions

Compounds 4-hydroxy benzoic acid and -D-Glucitol fatty acid esters have been successfully synthesized and characterized by FTIR. Their potential as surface active agents compounds have been proved by reducing surface tension and saturated vapor pressure of Iraqi gasoline at more than one temperature, making their use possible to reduce the evaporation of gasoline and thereby reduce pollutants in the environment as well as reducing economic losses.

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