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Changing of Electrical Conductivity, Acidity Function Voltage of Potential Difference for Salicylic Acid and Substituted to Study Adsorption Mechanism on Alumina Surface and the Factors Affecting it

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ABSTRACT

In this study and for the first time there are the use of the specific electrical conductivity, acidity function, difference potential for salicylic and acetyl salicylic acids (Aspirin) solution before and after adsorption to describe and elicit adsorption mechanism as followed :-

$MO^{-} + H_2O \rightarrow MOH$ (first step)

$MOH + H^{+} \rightarrow MOH_2^{+}$

MOH_2^{+} + Anion $L^{-} \rightarrow MOH_2^{+}\text{--------}^{-}L$ (second step)

Mo^{-} (alumina surface) , ^{-}L (anione)

The adsorption mechanism is represented by two steps the first is to change the charge of the alumina surface from the negative polarized charge to the positive polarized charge by adsorption of hydrogen ions and water molecules from the acid solution to the surface of the alumina by adsorption and the second step represented by the adsorption of the negative anion of the acid to the surface. Salicylic acid give (H^{+}) and salicylate ions (anione) and the presence of hydroxyl group on ortho position helps in the stabilization of anion and increase acidity compared with acetyl group. The adsorption process leads to an increase of acidity function by means of the decrease of (H^{+}) ions in the solution by transition from solution to surface of alumina. This is the first step of adsorption and the second step represented by the transition of salicylate ions. This in turn causes decrease of difference potential between the acidic solution and alumina surface. Decrease of (H^{+}) ions in solution causes decrease in electrical conductivity of solution. All these changes clarify mechanism of ions transition and their type from the solution to surface.

1. INTRODUCTION

The salicylic acid (SA) and o-acetyl Salicylic (OACSA) acid are important compounds used widely in pharmaceutical industries [1] .The (SA) producing intra hydrogen bonding gives stabilized molecule [2]. The adsorption of organic molecules on alumina surface gives physical and chemical adsorption and evidence of interaction of phenolic group with surface by a little hydrogen bond [3]. The adsorption of salicylate ions on alumina increases adsorption of molecules because of increasing the density of π electrons on the center of adsorption [4].

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The main mechanism of adsorption includes an electrostatic interaction between ions and surface formation hydrogen bonding to producing a complex surface [5] [6]. The activated alumina contains numbers of water molecules, the adsorption occurs on the surface of alumina by (AL- O^{-}) bond and the acidic position. The presence of (H^{+}) ions which is produced from ionized acid and water as solvent interact with surface changing charges of alumina surface and the oxide/water interface happens as follows:

$MO^{-} + H_2O \rightarrow MOH$

$MOH + H^{+} \rightarrow MOH_2^{+}$

MOH_2^{+} + Anion $L^{-} \rightarrow MOH_2^{+}\text{--------}^{-}L$

The chemical reaction occurring at the surface (oxide-water interface) is agreat importance as solid-solution interaction by adsorption which changing the electrical charges system for alumina surface the presence of ions in the solution with charges and apolar charges on alumina causing difference in potential between the surface and solution[7]. The adsorption process affects on the acidity function of solution while the acidity function changing the decrease or increase of (H^+) ions in solution. There are a relation between changing the electrical properties and adsorption process which gives describe transition of ions from solution to surface by means adsorption mechanisn and always the charges try to transition until its arrival to equilibrium.The aim of this study is using changing of electrical properties by adsorption describe the mechanism of adsorption process.

2. MATERIALS AND METHODS

1- Chemical materials: The chemical processing from BDH, Fluka companies, which are used without any purification

Compounds	Symbols
A- Salicylic acid	(SA)
B- O-Acetyl Salicylic acid	(OACSA)
C- Aluminum oxide	(Alumina)
D- Ethanol	
E- Distilled water	
F- Sodium hydroxide	
J- Potassium hydrogen phthalate	
H - pH.. indicator	

2- Instruments:

A- Shaker with water bath/Julabo sw23 (100 cycle/min).

B-Electrical oven/memert.

C-pH meter/HANNA instruments. Code pH 211SIN: 52058I, LOT11061, Romania.

D-Hanna instruments, Multi-Range conductiuity meters code: EC214, Romania.

3-Use one batch method to study the adsorption process.

4-Determination of carboxylic acids by titrometric method.

5-Use 1gm of alumina as adsorbent substance.

6-All the measurements produced at the nature acidity function for each acid, because the adsorption of carboxylic acids has previously been noticed to be strongest when the solution (pH) at nature acidity function.

7-Contact time:

Acid	Contact time	% adsorption
SA	30 min	87.2
OACSA	30 min	78.0

8-Different temperatures used with range: (293,303,313,323,333 K°)

Acid	Temperature	% adsorption
SA	333	92.0
OACSA	293	79.0

9-Use symbols instead of words as follows:

A- Conc. (M) = Concentration of acid solution. (Molarity).

B-Temp = Temperature used in our study.

C- pH, B.A = acidity function before adsorption.

D- pH,A.A= acidity function after adsorption.

E-MV, B.A= Difference potential millivolt before adsorption.

F-MV, B.A= Difference potential millivolt after adsorption.

G-SP.C, B.A= Specific conductivity ($s.cm^{-1}$) before adsorption.

H-S.P.C, A.A= Specific conductivity (s.cm) after adsorption.

I- % ads= present age adsorption.

10- All acids solvated by using a mixture solvent of 95% distilled water and 5% Ethanol.

3. RESULTS AND DISCUSSION

1-Salicylic acid ionized to give (H^+) and (salicylate) ions producing intra hydrogen bonding and adsorbed more radially to alumina surface from solution [2]. The results in table (1) show an increase in (pH) values after adsorption. This means a decrease of (H^+) ions in solution which are the transitions to adsorbed on the surface of alumina which carried a negative polar charges, the water molecules sharing (H^+) ions with the first step of adsorption to producing a layer or more than one layer converts the surface from a negative polar charges to a positive polar charges. Alumina surface interacts with water by presence of (H^+) ions producing of a hydroxyl sites, because the surface has amphoteric behavior. The second step of adsorption includes a transition of salicylate ion which carries negative charges to surface, the presence of (OH) group in ortho position producing hydrogen bonding between oxygen of carboxyl group and hydroxyl in ortho position which has a little bulk obstruction contribute to formation of hydrogen bridge readily. All this happensd because the surface of alumina was effected by interactions between the solid-solution interphase. These transitions of ions cause difference in the values of (pH, Mv and Sp.c) after adsorption. The results in table (1) clarify the effect of concentration on physical properties before and after adsorption.

Table 1: Effect of concentration on the pH, MV, Sp.c and percentage of adsorption at 303K for salicylic acid.

Conc.(M)	pH.B.A	pH.A.A	Mv.B.A	Mv.A.A	Sp.c.B.A	Sp.c.A.A	%ads.
0.005	3.04	5.58	234.4	80.1	624	191.8	87.75
0.007	2.94	4.33	241.0	157.5	780	299	74.10
0.009	2.87	3.95	245.4	191.7	917	334	65.70
0.011	2.83	3.61	247.4	199.8	1014	409	61.42
0.013	2.78	3.53	250.8	208.1	1134	511	56.65

3.1 .Effect of Concentration on Changing of pH, MV, SP.C Values

The results before adsorption in table (1) refer to a decrease of pH values with increase of concentration while increasing of MV and SP.C. values because to an increase of (H^+) and salicylate ions in solution . Now we are showing a difference of these values which are mentioned after adsorption at all concentrations, the pH values increase this gives indicates a decrease of (H^+) ions in solution by transition to surface of alumina with water molecules. In the first step of adsorption, while we notes a high decrease of MV and SP.C values at all concentrations, This differences confirmed our pre-conclusions about transition (H^+) and salicylate ions by adsorption from solution to alumina surface. The increase of concentration causes increase on MV values between the surface and solution and increasing of SP.C values in solution, but the efficiency of adsorption decrease with increasing of concentration instead of available of ions in solution because of molecular interactions and association [8][9] between molecules and ions in solution with increasing of concentration cause decrease the transition of (H^+) and salicylate ions to surface, which affects directly on the adsorption percentage. The adsorption process causes loss of proton readily, the percentage of adsorption depend on deprotonation of acid which a relative easy process and transition of ions from solution to surface but increasing of interaction in solution by concentration decrease this process decreasing (%ads.).we are studying the difference in (pH, MV and SP.C) before and after adsorption at low concentration (0.005M) and at high concentration (0.013M) at 303 K°, The results listed in table (2). The difference of pH at low and high concentration before adsorption gives low values which refer to increasing of concentration of acid solution cause increase of interactions gives low values of (ΔpH) and (ΔMv) compared with values after adsorption but (ΔSp.C) value before adsorption more than this value after adsorption.

Table 2: The difference of (pH, MV and SP.C) from a low and high concentration from table(1) at 303 K°.

Before adsorption (0.005M→0.013M)			After adsorption (0.005M→0.013M)		
ΔpH	ΔMv	ΔSp.C/ $s.cm^{-1}$	ΔpH	ΔMv	ΔSp.C/ $s.cm^{-1}$
0.26	16.4	510	2.05	128	319.2

But when studying the effect of concentration on (pH, MV and SP.C) at the same concentration before and after adsorption at 0.005M alone and 0.013M at 303 K° it appeared as in table (3), Horizontal difference.

Table 3: The difference of (pH, MV and SP.C) between a low and high concentration from table(1) at 303 K°.

Difference before and after adsorption at 0.005m			Difference before and after adsorption at 0.0013m		
ΔpH	ΔMv	ΔSp.C/ $s.cm^{-1}$	ΔpH	ΔMv	ΔSp.C/ $s.cm^{-1}$
2.54	154.3	104.5	0.75	42.7	623

From table (3) the study of the effect of adsorption on acidic solution in presence of alumina surface at the same concentration (0.005M) we note a high difference on pH,MV and low values of SP.C because of a less presence of (H^+) and salicylate ions in solution after adsorption at (0.005M) which gives a high percentage of adsorption (87.75) while at (0.013M) after adsorption was obtained a low difference on pH,MV and with a high presence of (H^+) and salicylate ions in solution with a low percentage of adsorption (56.65) eventually the increase of concentration causes decreasing of transition of salicylate ions to alumina surface, which causes decreasing of adsorption percentage.

Effect of Temperature:

Generally the increase of temperature causes increase of ionization and removal of a molecular interaction and association which increases adsorption percentage. When we review table (4),at 0.005M of acid solution we notice that the increase of temperature from 293K° to 333K° for acid solution without presence of alumina gives low difference on pH values (ΔpH=0.41) and with presence of alumina (ΔpH =0.12). These values show that a little effect of temperatures gives more ions of (H^+), while the ΔMV value before adsorption (ΔMV=24.6).Here we notice a lower value for (ΔSP.C=0.87) before adsorption while we show a high (ΔSP.C) after adsorption between 293K° to 333K°. The adsorption percentage increases with increasing of temperature by low values. These results confirms that a low effect of temperature on adsorption process while a high effect on electrical conductivity[6][8][10].

Table 4: The effect of temperature on (pH, MV, SP.C) and %ads for salicylic acid at (0.005M).

Temp	pH B.A	pH A.A	Mv B.A	Mv A.A	Sp.C. B.A	Sp.C. A.A	%ads.
293	3.18	5.34	226.1	94.1	603	165.3	85.30
303	3.04	5.58	234.4	80.1	624	191.8	87.75
313	2.95	5.17	240.1	106.4	634	216	89.22
323	2.84	5.33	246.8	96.1	657	250	90.20
333	2.77	5.22	250.7	104.3	690	280	92.16

Table 5: Effect of temperature on pH, MV, SP.C and %ads for salicylic acid at (0.013M)

Temp	pH B.A	pH A.A	Mv B.A	Mv A.A	Sp.C. B.A	Sp.C. A.A	%ads.
293	2.85	3.53	245.9	203.7	1101	484	56.28
303	2.78	3.41	250.8	208.1	1134	511	56.65
313	2.60	3.38	256.8	217.3	1145	545	57.41
323	2.53	3.28	264.9	203.3	1167	564	57.78
333	2.41	3.24	271.3	213.3	1195	575	58.54

To clarify the effect of both concentration and temperature on changing of pH, MV and SP.C by adsorption, the results listed in tables (6) and (7),respectively.

Table 6: values of PH, MV and SP.C for salicylic acid before and after adsorption with different concentration at 303K°

Conc.M	pH /B.A	pH/A.A	Mv/B.A	Mv/A.A	Sp.C. B.A	Sp.C. A.A	%ads.
0.005	3.04	5.43	234.4	80.1	624	191.8	87.75
0.007	2.94	4.33	241	157.5	780	299	74.10
0.009	2.87	4.21	245.4	191.7	917	334	65.70
0.011	2.83	3.61	247.4	199.8	1014	409	61.42
0.013	2.78	3.53	250.8	208.1	1134	511	56.65

Table 7: values of pH, MV and SP.C for salicylic acid before and after adsorption with different concentrations at 333K°

Conc.M	pH /B.A	pH/A.A	Mv/B.A	Mv/A.A	Sp.C. B.A	Sp.C. A.A	%ads.
0.005	2.77	5.22	250.7	104.3	690	280	92.16
0.007	2.61	4.44	259.7	150.6	825	420	77.95
0.009	2.51	3.93	265.6	180.8	959	470	70.06
0.011	2.49	3.67	268.2	198.8	1088	510	63.92
0.013	2.41	3.38	271.3	213.3	1195	575	58.54

2- Acetyl Salicylic acid: In this acid the hydroxyl group in salicylic acid is exchanged by acetyl group for these reasons here is no intra hydrogen bonding in molecule and the ionization less than salicylic acid and have a steric effect compared with hydroxyl group.

A-Effect of concentration: when focusing on the results in table (8), we will show B.A the values of pH and MV are not affected clearly with increasing of concentration of acid solution while a clear difference in values of SP.C and these values at a high concentration (0.013M) do not equal half value of SP.C of SA at a lower concentration (0.005M).

Table 8: The difference of values pH, MV, SP.C and %ads with increasing of concentration BA and A.A for OACSA at 303K°

Conc.M	**pH /B.A**	**pH/A.A**	**Mv/B.A**	**Mv/A.A**	**Sp.C. B.A**	**Sp.C. A.A**	**%ads.**
0.005	3.3	4.61	218.8	138.9	308	170	69.04
0.007	3.24	4.29	223.3	159.3	380	236	52.71
0.009	3.16	4.04	227.3	172.9	434	261	43.22
0.011	3.10	3.90	231.0	182.4	486	298	36.91
0.013	3.06	3.77	233.0	191.6	532	336	30.85

In presence of metal oxide we notice an increase of pH values with a low difference at all concentrations for example at low concentration ($\Delta pH=1.31$) while at high concentration ($\Delta pH=0.71$). This means a viability of (H^+) ions and Carboxylate ions in solution at lower concentration gives high percentage because the ions have free motion and transition easily to surface compare with solution at a high concentration, which decrease of transition of ions to surface and stays in solution ,for these reasons the %ads decrease as shown in table (8).

To compare between the activity of concentration and temperature on adsorption process at the same concentration the results tabulated in table (9).

Table 9: The difference of values pH, MV, SP.C and %ads with increasing of concentration BA and A.A for OACSA at 333K°

Conc.M	pH /B.A	pH/A.A	Mv/B.A	Mv/A.A	Sp.C. B.A	Sp.C. A.A	%ads.
0.005	3.05	4.36	234.7	173.3	318	260	77.0
0.007	2.94	3.73	239.9	192.6	391	332	60.71
0.009	2.87	3.55	244.3	204	444	397	52.22
0.011	2.82	3.42	248.3	212.6	507	430	42.73
0.013	2.76	3.32	250.6	215.3	556	506	38.85

The results in table (9) refers to the increasing of temperature to 333K° leads to decreasing of (pH) values before and after adsorption by means increase of (H^+) ions in solution and increasing of (Mv.SP.C) values before and after adsorption compered with this values in table (8), because increasing of ions in solution happened by increasing of temperature from 303K° to 333K° by means addition a further energy to adsorption system causing increasing of %ads.

Generally there are decreasing of pH values with increasing of concentration, B.A as well as the MV values increased with small values but with a high values of SP.C , for OACSA while A.A the pH values increase by transition of (H^+) ions from solution to alumina, and the lower presence of (H^+) and Carboxylate ions affected on a conductivity values which decreased and lowered compared with this values for salicylic acid , the Mv values decreased too compared with these values B.A. Instead of increasing of ions in solution by increasing of concentration the ads. Percentage decreases continuously, we are reach to a conclusion that presence of acetyl group instead of hydroxyl group affects negatively at all the electrical values as well as %ads. converts from 69.4% at (0.005M) to 30.85% at (0.013M) as clarify in table (8).The another effect of concentration is the difficulty of moving of ions in solution and a less transition from solution to oxide surface, the steric effect of acetyl group in anion sharing this difficulty and each ion adsorbed on surface covered a several positions on surface shielding this position and it is preventing other ions from adsorbing on this position by effect called umbrella [15].

3.2. Effect of Temperature

Generally we are know the increasing of temperature causes increasing of adsorption, but our study about (OACSA) whose results listed in table (10), before adsorption the increasing of temperature causes a few of decrease of pH values with a few increase of MV and SP.C values. This means a less effect of temperatures on these values, but after adsorption, we noticed a small increasing of pH, small lowered in MV and a high decreasing of SP.C, From these results we can arriving to conclusion that a low producing of (H^+) and salicylate ions and transition of a few ions to surface cause lowering in MV values, and a high decreasing of SP.C at 293 K° , but a lower decreasing at 303 K° since SP.C at (293) K° = (157) while SP.C at (303)K° = (58) and % ads, decreased from 97% to 69.04%. This is because at 293k a further ions transition to surface which leads to decrease of SP.C in solution because decreasing of ions by adsorption but the increase of temperature to 303 K° cause leaving of ions from surface to solution which increases number of ions in solution which increasing the SP.C values leads to decreasing of %ads.

Now when combining the effect of concentration and temperature together on pH, MV SP.C and %ads we can say that instead of increasing of concentration and temperature cause increasing ionization and gives a further positive and negative ions by small values which increase MV and SP.C in solution comparing with charges on surface both the concertation and temperature have a negative effect on adsorption process but a positive effect on the numbers of ions in solution. From table (10) the increasing of temperature at (0.005)M cause increasing of (H^+) and salicylate ions in solution B.A because of decreasing of (pH) values and increasing of Mv and SP.C with small values but A.A the (pH) value increase at all temperature and this values it self decreased with temperature while Mv and SP.C values decreased compared with this value B.A but the increase of temperature on adsorption process leads to increase at M.v and SP.C values it self leads to process at another ions stay in solution followed with decreasing of %ads with a few a values by means the transition of ions happened but still other ions presence in solution do not helps to obtains increase of %ads. This increase of ions happens because of the increasing of temperature which leads to leaves of adsorbent ions the surface to solution increase Mv and SP.C values decreasing of %ads.The results agreement with [11-13].

Table 10: Effect of temperature on pH, MV and SP.C, %ads for OACSA at 0.005M

Temp.k°	pH. BA	pH A.A	Mv B.A	Mv A.A	Sp.C. B.A	Sp.C. A.A	%ads.
293	3.44	4.61	210.1	125.8	307	150	79

303	3.30	4.34	218.8	138.9	308	170	77
313	3.23	4.30	223.4	147.1	310	190	74.96
323	3.13	4.17	229.5	155.3	315	214	72.56
333	3.05	4.06	234.7	173.3	318	260	69.04

Table 11: Effect of temperature and concentration on pH, MV and SP.C values for OACSA at (0.013M)

Temp.k°	pH /B.A	pH/A.A	Mv/B.A	Mv/A.A	Sp.C. B.A	Sp.C. A.A	%ads.
293	3.12	3.96	230.5	188.4	520	322	39.62
303	3.06	3.75	233.0	191.6	532	336	38.85
313	2.96	3.56	239.4	193.9	538	357	35.38
323	2.86	3.53	245.3	202.6	540	362	33.92
333	2.76	3.32	250.6	215.3	556	506	30.85

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تغير التوصيلية الكهربائية، الدالة الحامضية وفرق الجهد لحامض السلسليك ومعوضه لدراسة ميكانيكية الامتزاز على سطح الالومينا والعوامل المؤثرة عليها.

خليل ابراهيم النعيمي

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الخلاصة:

في هذه الدراسة ولأول مرة تستخدم كل من التوصيلية الكهربائية النوعية، الدالة الحامضية وفرق الجهد (لمحلولي) حامضي السالساليك والاستيل سالساليك (الاسبرين) قبل وبعد الامتزاز لوصف واستنباط ميكانيكية الامتزاز وبالشكل الاتي:

$MO^{-} + H_2O \rightarrow MOH$ (first step)

$MOH + H^{+} \rightarrow MOH_2^{+}$

$MOH_2^{+} + \text{Anion } L^{-} \rightarrow MOH_2^{+} \text{-------} {}^{-}L$ (second step)

Mo^{-} (alumina surface) , ^{-}L (anione)

ان ميكانيكية الامتزاز تتمثل بخطوتين الأولى هي تغيير شحنة سطح الالومينا من شحنه مستقطبة سالبة الى شحنة مستقطبة موجبه بواسطة ايونات الهيدروجين وجزيئات الماء من خلال امتزازهما من المحلول الحامضي الى سطح الالومينا , والخطوة الثانية المتمثلة بامتزاز الانيون السالب للحامض على السطح .ان حامض السالساليك يعطي ايون (H^{+}) والسالساليت (الانيون), ان وجود مجموعة الهيدروكسيل في الموقع اورثو تساعد في استقراريه الانيون تزيد من الحامضية بالمقارنة مع مجموعة الاستيل ،ان عملية الامتزاز تؤدي الى زيادة الدالة الحامضية بسبب انخفاض عدد ايونات (H^{+}) في المحلول بانتقالها من المحلول الى سطح الالومينا وهذا ناتج الخطوة الأولى لعملية الامتزاز والثانية تمثلت بانتقال ايونات السالساليت وهذا بدوره يؤدي الى اختلاف في الجهد بين المحلول الحامضي وسطح الالومينا ,ان انخفاض ايونات (H^{+})كذالك يسبب انخفاض التوصيلية الكهربائية للمحلول كل هذه التغيرات دراستها توضح ميكانيكية انتقال الايونات ونوعها من المحلول الى السطح .

الكلمات المفتاحية: *توصيلية كهربائية، دالة حامضية، فرق جهد، حامض سالساليك، سطح الومينا .*