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Analytical studies on anodic ethoxylation of salicylic acid

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Abstract
Electro organic chemistry is a multidisciplinary science overlapping the vast fields of organic chemistry, biochemistry, physical chemistry and electrochemistry. The electrochemical oxidation of aromatic compounds on graphite and platinum electrode in alcoholic media has observed to yield ethers or acetals as a sequence of addition or substitution by alkoxy groups. This research work is having the major stress on electro analytical aspects of salicylic acid. These electro analytical studies include (i) cyclic voltammogram, (ii) working electrode variation studies (iii) pH variation studies (iv) scan rate variation studies and (v) multiple scan studies with an intention to explore whether the envisaged anodic process is possible or not. From the analytical studies, the oxidation potentials, favorable pH condition for the ethoxylation and also the formation of polymer on the working electrodes are predicted.

Keywords: cyclic voltammetry, working electrode, ethoxylation, electro analytical studies

1. Introduction
This research work is having the major stress on electro analytical aspects of salicylic acid. Salicylic acid (SA) is a phenolic phyto hormone and is found in plants with roles in plant growth and development, photosynthesis, transpiration, ion uptake and transport. SA also induces specific changes in leaf anatomy and chloroplast structure. SA is involved in endogenous signaling, mediating in plant defense against pathogens [3].
Among all other electrochemical processes, analogues to the corresponding chemical routs, anodic oxidations of aliphatics, aromatics, heterocyclic, etc., either directly from a major part of electro organic research [4-6]. Electrochemical hydroxylation, alkoxylation, acyloxylation, cyanation and halogenations are some of the important functionalization reaction exhaustively performed during the past three decades [7-9]. In the normal procedure of manufacturing of certain pharmaceutical and industrial compounds, alkoxy compounds are found to be employed as source generator [10]. Henceforth, the electrochemical alkoxylation of aromatic compounds has evolved as a spontaneous choice for the current project.
The electro chemical oxidation of aromatic compounds on graphite or platinum electrode in alcoholic media has observed to yield ethers or acetals as a sequence of addition or substitution by alkoxy groups. The present study encompasses various aspects of the electro analytical studies on electrochemical ethoxylation of Salicylic acid.
In this present work, platinum and glassy carbon electrodes are taken as working electrodes. The electrode potentials of working electrodes are found out by taking Ag/AgCl electrode as reference. The ethoxylation of SA was carried out in ethyl alcohol medium. KOH, KCl, H_2SO_4 were used as supporting electrolytes in alkaline, weakly acidic and strongly acidic conditions respectively. The work is carried out to predict the anodic peak potentials at different pH media, to find out whether the reaction is diffusion or adsorption controlled and to ascertaining the formation of any polymer films on the surface of the working electrode.

2. Materials and Method
2.1 Apparatus
Voltammogramms are recorded with potentiostat CH 10 (Sinsil international) interfaced to 663 VA stand (Metrohm) and Sync Master B1930 computer. A three electrode configuration is used with platinum / glassy carbon electrode as the working electrode, a silver- silver chloride reference electrode and a platinum electrode wire as the auxiliary electrode. The working electrode was pretreated by polishing it with an alumina – water slurry followed by washing in an ultrasonic path.

2.2 Reagents and solutions

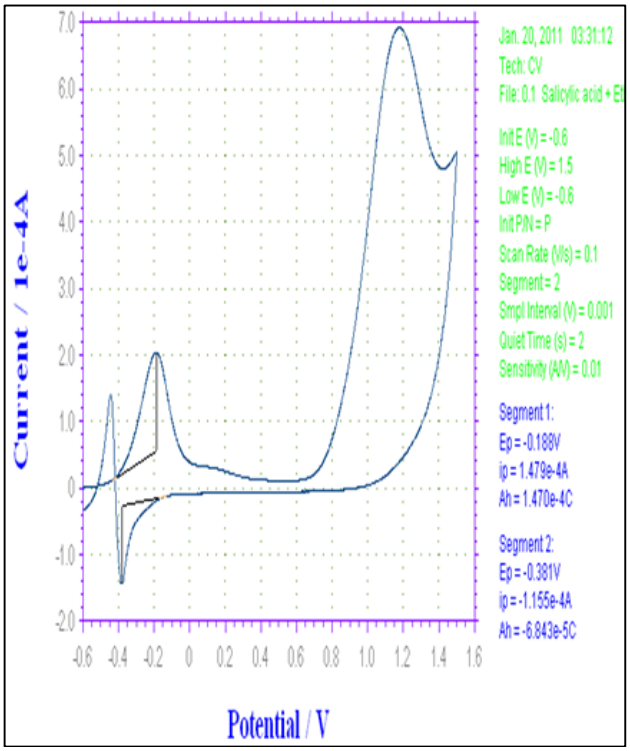
All reagents are of analytical reagent grade and ultra pure water is used throughout 0.001 M Salicylic acid, 1M H_2SO_4/KOH/ KCl, 0.5M Ethanol were prepared freshly. The pH of the different reaction mixtures measured with pen type pH meter. The solutions are stored in a light protected cool location.

2.3 Methodology

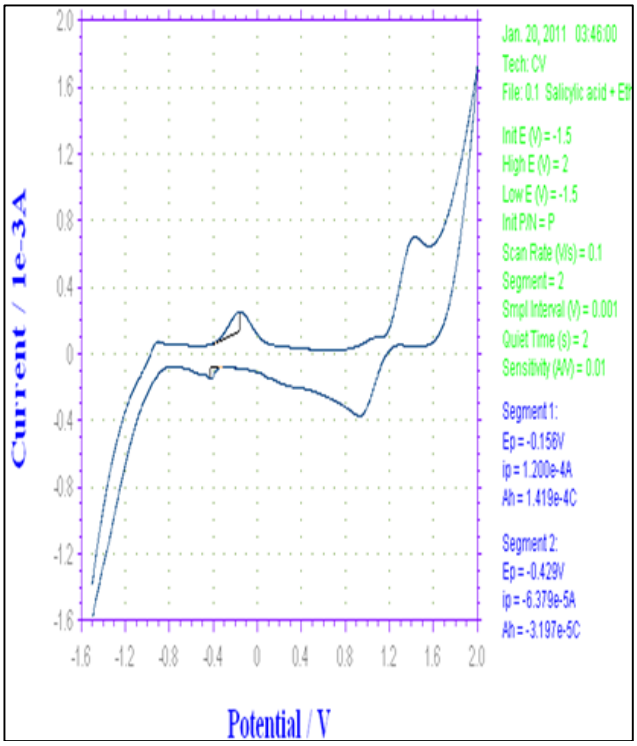
The three electrode system with platinum/glassy carbon electrode as the working electrode, platinum wire as the auxiliary electrode and Ag/AgCl electrode as the reference electrode is constructed in an undivided cell. In order to change the pH of the system 1M solutions of H_2SO_4/KOH/ KCl are taken. These solutions are also working as the sources of supporting electrolytes. Then the cylclic voltammogram is recorded with different scan rates, different working electrodes and different pH conditions. To arrive at an idea about the polymerization of SA on working electrode multiple scan cyclic voltammogram was also recorded.

3. Result and Discussion

3.1 Cyclic voltammetric analysis



salicylic acid+ethanol+KOH



salicylic acid+ ethanol + KCl

From the above cyclic voltammograms, the following anodic otentials and anodic currents are observed

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S No	Substrate mixture (Working electrode, Pt)	pH	Anodic potentials (volt)		Anodic current (mA)	
			Peak1	Peak2	Peak 1	Peak 2
1.	Salicylic acid + Ethanol + KOH	12.6	-0.1854	1.1788	2.301	6.887
2.	Salicylic acid + Ethanol + KCl	3.10	-0.156	1.416	2.523	7.206

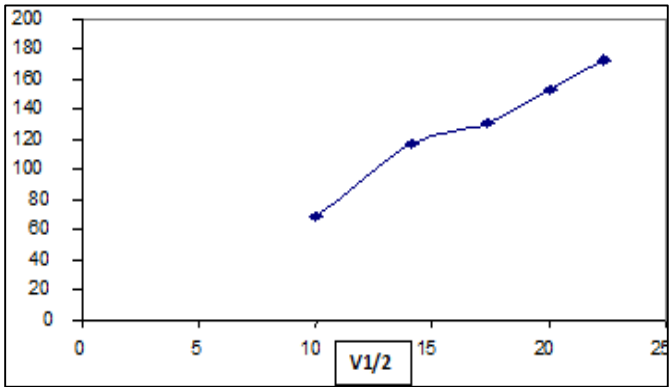
3.2 Scan rate variation studies

Cyclic voltammograms for the ethoxylation mixtures at acidic basic and neutral media are recorded with variable sweep rates along the range of 100/500 mV/second.

3.2.1 Salicylic acid +Ethanol + KOH

Substrate: 0.001 M [Alcohol]: 0.1m Anode: Pt Cathode: Pt

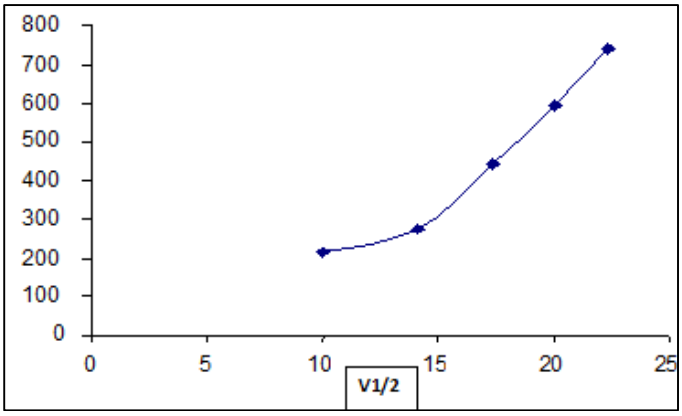
Parameters	Scan rate v (mV/s)				
	100	200	300	400	500
v ½	10	14.14	17.32	20	22.36
Ip (µA)	68	117	131	153	173
Ep (v)	1.17	1.24	1.27	1.31	1.73



3.2.2 Salicylic acid +Ethanol + KCl

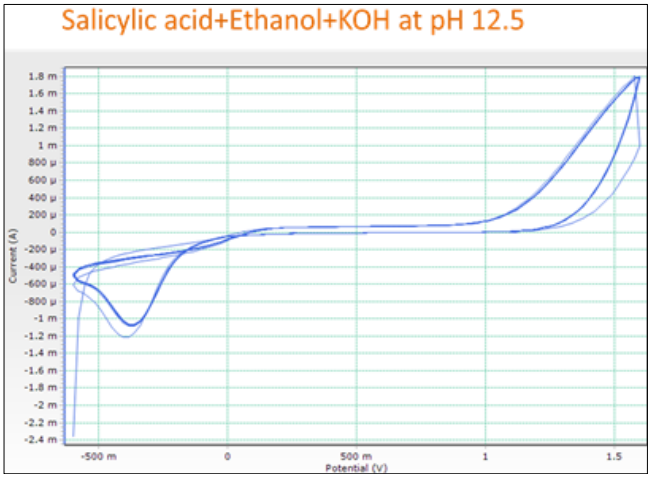
Substrate: 0.001 M [Alcohol]: 0.1m Anode: Pt Cathode : Pt

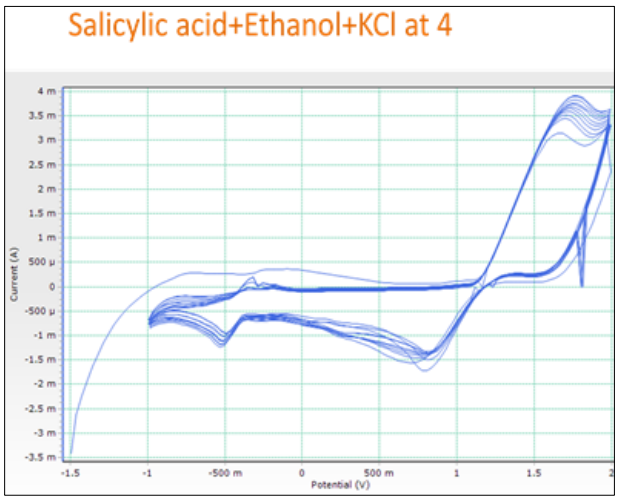
Parameters	Scan rate v (mV/s)				
	100	200	300	400	500
v ½	10	14.14	17.32	20	22.36
Ip (µA)	252	383	410	475	574
Ep (v)	-0.156	-0.136	-0.130	-0.124	-0.130



From the scan rate variation studies, it is observed that the ethoxylation process under the expected conditions are diffusion controlled.

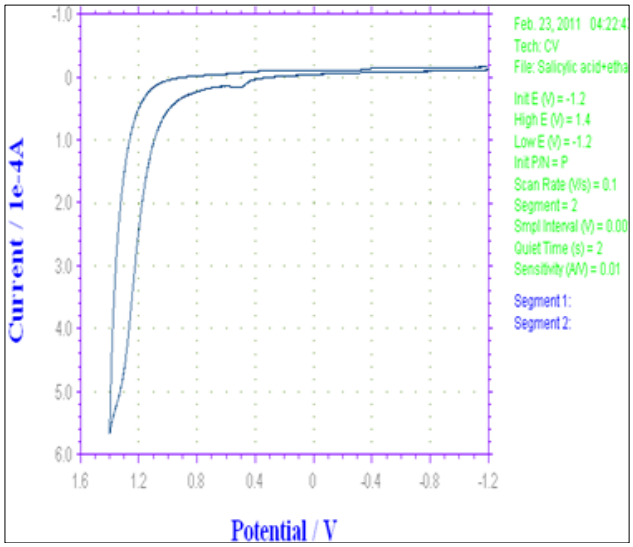
3.3 Multiple Scan Studies



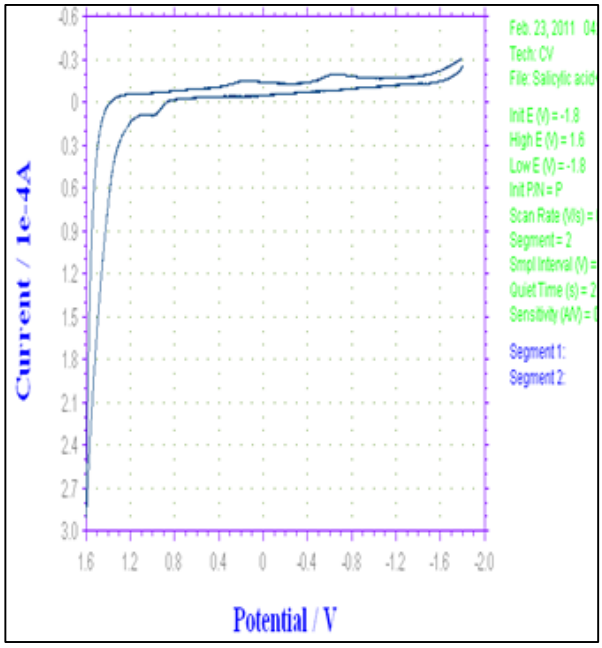


3.4 Electrode Variation Studies

The following cyclic voltommograms obtained by using the working electrode as glassy carbon electrode.



Salicylic acid +Ethanol+KCl



Salicylic acid +Ethanol +KOH

The following anodic potentials and anodic currents are obtained by using glassy carbon electrode as the working electrode.

S. No	SUBSTRATES (Working electrode-Glassy carbon)		pH	Anodic potentials (volt)		Anodic current (mA)	
			Peak1		Peak2	Peak 1	Peak 2
1	Salicylic acid + Ethanol + KOH	12.6	0.5102		-	1.607	-
2	Salicylic acid + Ethanol + KCl	3.10	-0.3190		0.992	6.768	9.024

4. Conclusion

- The following are the conclusion drawn from the Electroanalytical studies on ethoxylation of salicylic acid.
- The electro analytical studies carried out with Pt & Glassy carbon as the working electrode, the possible anodic potentials for the electro chemical ethoxylation of salicylic acid are found out.
- It is also observed that the working potentials vary with Pt and Glassy Carbon.
- By changing the pH of the reaction medium the favorable pH for the ethoxylation of salicylic acid are predicted.
- By the scan rate variation studies it is also observed that the ethoxylation of salicylic acid is diffusion controlled and not adsorption controlled.
- The multiple scan rate studies for ethoxylation of salicylic acid revealed that no polymer coating during the electrolysis.

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