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***THE ELECTROCHEMICAL OXIDATION  
OF HYDROGEN PEROXIDE  
ON NICKEL ELECTRODES  
IN PHOSPHATE BUFFER SOLUTIONS***

A thesis presented in partial fulfilment of the requirements for  
the degree of Master of Science in Chemistry  
at Massey University, Palmerston North, New Zealand

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2000

## ABSTRACT

The electrochemical oxidation of hydrogen peroxide was studied at nickel electrodes in phosphate buffer solutions. This reaction is of interest because of its possible use in the construction of devices for the electrochemical detection of hydrogen peroxide. The devices developed could be advantageous in many industrial and medical processes.

Using the electrochemical technique, staircase potentiometry, the activity of the nickel electrode in catalysing hydrogen peroxide oxidation was evaluated over a range of bulk hydrogen peroxide concentrations, rotation rates, electrode potentials, temperatures, buffer concentrations and pH.

A mechanism was developed to account for the observed activity. This was based on a previous model developed for the electrochemical oxidation of hydrogen peroxide at platinum electrodes [1-6]. The mechanism involved  $\text{H}_2\text{O}_2$  interaction with binding sites on the surface of the electrode. These were initially identified as a nickel phase oxide,  $\text{Ni}(\text{OH})_2$ . Later, the involvement of buffer phosphate species  $\text{HPO}_4^-$  was identified. Hydrogen peroxide is adsorbed onto the binding site to form the complex  $\text{Ni}_{\text{BS}}\cdot\text{H}_2\text{O}_2$ . This complex then undergoes an internal charge transfer to form a reduced nickel site, liberating the products water and oxygen. The binding site regenerates electrochemically to give rise to an amperometric signal with the release of protons. A side reaction was proposed which involved an interaction between the binding sites and dioxygen. This interaction competitively inhibited the binding of  $\text{H}_2\text{O}_2$ .

A rate equation was derived to account for all the surface sites involved in the proposed reaction mechanism. The kinetic, equilibrium and thermodynamic constants of the resulting model were optimised by a SIMPLEX procedure. These constants were in turn used in conjunction with the rate equation to produce synthetic responses, which were then compared to the observed steady-state response. A satisfactory fit was found over the entire range of conditions studied. This supported the proposition that the mechanism was appropriate.

The equilibrium constants were found to be potential invariant, with  $K_1 = 4.43 \times 10^{-3}$  and  $K_4 = 0.360 \text{ m}^3 \text{ mol}^{-1}$  at  $20^\circ\text{C}$ . The former,  $K_1$ , was exothermic, with  $\Delta H = -28.32 \text{ kJ K}^{-1}$  between 5 and  $25^\circ\text{C}$ , and became significantly more exothermic, with  $\Delta H = -198.33 \text{ kJ K}^{-1}$

between 25 and 35°C. In contrast,  $K_4$  was slightly endothermic, with  $\Delta H = -16.5 \text{ kJ K}^{-1}$  over the temperature range. One rate constant could be approximated to be potential invariant,  $k_3N = 7.99 \times 10^{-4} \text{ mol m}^{-2} \text{ s}^{-1}$  at 20°C. Whereas, the other,  $k_2N$ , varied with potential. Both rate constants were endothermic with pseudo-activation energies for  $k_3N$  being  $24.3 \text{ kJ mol}^{-1}$  and for  $k_2N$  ranging between  $130\text{--}80 \text{ kJ mol}^{-1}$  (depending on electrode potential).

An optimum pH region for the study of  $\text{H}_2\text{O}_2$  oxidation at nickel was found to be between pH 4 and 9. Above and below these bounds competitive reactions occurred that were not attributable to hydrogen peroxide and insignificant rates of reaction for electrochemical measurement were found.

The phosphate species  $\text{HPO}_4^{-2}$  was identified as being involved in the oxidative mechanism. The nature of this involvement was complex, with  $\text{HPO}_4^{-2}$  both inhibiting and facilitating  $\text{H}_2\text{O}_2$  oxidation, depending on surface concentration. To accommodate this, the proposed mechanism was further modified to include this involvement. It was proposed that  $\text{HPO}_4^{-2}$  was required to form the  $\text{H}_2\text{O}_2$  binding site from a nickel precursor site on the electrode surface. However, the complexation of a second  $\text{HPO}_4^{-2}$  to this site would inhibit  $\text{H}_2\text{O}_2$  binding.

The work presented in this thesis represents a fundamental study into the electrochemical behaviour of hydrogen peroxide at nickel electrodes. This behaviour has been clearly identified over a range of temperatures, hydrodynamic conditions, buffer compositions and concentrations. This enabled a new and comprehensive mechanism, for the oxidation of hydrogen peroxide at nickel electrode, to be developed.

## ACKNOWLEDGEMENTS

First I would like to acknowledge the supreme efforts of Simon without whom this work would have been impossible. I would like to thank him for his time, patience, guidance, and humour in teaching me an area of chemistry in which I had no prior experience. You always made time for the most inane of questions and seldom lived up to your middle initial - one could not ask for a better supervisor.

To my co-workers and friends Giovanna, Ant, Emad, and Michael you made an inspired lab. Emad (the father figure) - your help and guidance were greatly appreciated, I loved our talks. Michael (lunch!!) you contributed so much to the humour of the lab. G and Ant I could not ask for better friends, you most of all made doing Masters a blast. Thanks.

To Gav, Jo (meeeow), tall Paul, Sonya, Rekha, big Paul, Steve, Ester, Adrian, Andrew, Wayne, Garf, farmer Jim, G (respect my authoritar), Ant & Bec, Clem, Dave, and Beccy (I hope I didn't miss anyone). Thank you for being social and getting involved, Viva Viagra, need I say more? Your fantastic friendship and humour has made my life so much more colourful (in a good way). Beccy, despite my complaints, your gentle nagging and all that you have given me are greatly appreciated. To the other chemistry students, staff (esp. Dave, Andy and Emily), and other affiliated members your easy going friendly attitude made life so much better.

Last but certainly not least I wish to acknowledge my family. Mum, Dad, Charles & Shelley, Granny, Lek and all the cousins, uncles, and aunts who have given me so much love, never held me back, for always encouraging me and letting me believe I can do anything. It makes it so much easier knowing you are always there. I am always thinking of you.

I have had so much fun and have many wonderful memories it saddens me that I have to finish.

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# LIST OF SYMBOLS

<u>Symbol</u>	<u>Description</u>	<u>Unit</u>
$\alpha_i$	hydrodynamic radii for species $i$	$\text{m}^2 \text{s}^{-1}$
$c$	concentration	$\text{mol L}^{-1}$
$d_{\text{av}}$	average deviation	$\text{mA cm}^{-2}$
$D$	diffusion coefficient	$\text{m}^2 \text{s}^{-1}$
$D_i$	diffusion coefficients for species $i$	$\text{m}^2 \text{s}^{-1}$
$e^-$	electron	
$E$	electrode potential	$\text{mV}$
$E^\circ$	standard electrode potential	$\text{mV}$
$F$	Faraday constant	$\text{C mol}^{-1}$
$\Delta H_{\text{DBE}}$	dissociative bond energies	$\text{kJ mol}^{-1}$
$\Delta H$	enthalpy change	$\text{kJ mol}^{-1}$
$\Delta H^\circ$	enthalpy of formation	$\text{kJ mol}^{-1}$
$\Delta H^\ddagger$	activation energy	$\text{kJ mol}^{-1}$
$i$	current density	$\text{mA cm}^{-2}$
$i_a$	anodic current density	$\text{mA}$
$i_c$	cathodic current density	$\text{mA}$
$i_{\text{obs}}$	current density observed	$\text{mA cm}^{-2}$
$i_L$	limiting current density	$\text{mA cm}^{-2}$
$i_k$	kinetic current density	$\text{mA cm}^{-2}$
$i_{\text{calc}}$	current density calculated	$\text{mA cm}^{-2}$
$I$	current	$\text{mA}$
$j$	flux or rate of diffusion	$\text{mol m}^{-2} \text{s}^{-1}$
$k_2$	heterogeneous rate constant	$\text{m s}^{-1}$
$k_3$	heterogeneous rate constant	$\text{m s}^{-1}$
$k_D$	diffusion constant	$(\text{m}^2 \text{s}^{-1})^{1/6}$
$k_f$	heterogeneous electron transfer rate constant	$\text{m s C}^{-1}$
$K_N$	equilibrium constant	$\text{m}^3 \text{mol}^{-1}$



$K_1$	equilibrium constant	$\text{m}^3 \text{mol}^{-1}$
$K_4$	equilibrium constant	$\text{m}^3 \text{mol}^{-1}$
$K_5$	equilibrium constant	$\text{m}^3 \text{mol}^{-1}$
$l$	length	m
$\bar{m}$	molarity	$\text{mol Kg}^{-1}$
$M$	molarity	$\text{mol L}^{-1}$
$n$	number of electrons	
$n_p$	number of data points	
$N$	number of binding sites	
$N^*$	number of precursor sites	
$r$	radius	m
$r_s$	sum of residuals	
$R_1/R_2$	roots of a quadratic	
Re	Reynolds number	
$\gamma$	mean activity coefficient	
$\eta$	Absolute viscosity	$\text{kg m}^{-1} \text{s}^{-1}$
$\theta$	surface coverage	
$\lambda_1/\lambda_2/\lambda_3$	roots of a 3 <sup>rd</sup> order polynomial	
$\nu$	kinematic viscosity	$\text{m}^2 \text{s}^{-1}$
$\rho$	density	$\text{g ml}^{-1}$
$\upsilon$	characteristic velocity	$\text{m}^2 \text{s}^{-1}$
$\omega$	angular velocity	$\text{rad s}^{-1}$

## LIST OF ABBREVIATIONS

BAS	Bioanalytical Systems
CE	counter electrode
CPE	carbon paste electrode
CV	cyclic voltammetry
DNA	deoxyribose nucleic acid
ECE	electron-transfer/chemical/electron-transfer mechanism
FIA	flow-injection analysis
GOD	glucose oxidase
H <sub>2</sub> O <sub>2</sub>	hydrogen peroxide
IFS	Institute of Fundamental Sciences
NADH	nicotinamide adenine dinucleotide hydride
NEXFAS	near edge x-ray adsorption fine structure
ppm	parts per million
RDE	rotating disc electrode
RE	reference electrode
rpm	revolutions per minute
SAM	self-assembled monolayers
SCE	saturated calomel electrode
SCP	staircase potentiometry
SHE	standard hydrogen electrode
WE	working electrode
wt/wt	weight by weight