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A Heterogeneous precipitate based sodium ion membrane sensors-Its preparation and analytical application

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Abstract

A heterogeneous precipitate have been used as ion carriers for the preparation of Na(I) selective membrane sensor. This electrode give near-nernstian responses in the linear concentration range of 1M to 1×10^{-5} M with detection limits of the order of 10^{-5} M. The stable potentiometric signals are obtained with in a short time period of 4 minutes.The effect of pH and the effect of medium have been studied found to give a better response selectivity coefficient values (log $K^{pot}_{Na,M}$) have been evaluated using fixed interference method.The sensor have also been used as indicator electrode in commercial products.

Key words: A Heterogeneous precipitate, Na(I) ion membrane sensor, fixed interference method.

Introduction

Sodium hydroxide is a caustic metallic base. It is used in many industries,mostly as a strong chemical base in the manufacture of pulp and paper,textiles,drinking water,soaps and detergents and as a drain cleaner.

Food uses of sodium hydroxide include washing or chemical peeling of fruits and vegetables, chocolate and cocoa processing, caramel coloring production,poultry scalding and soft drink processing.

The field of ion-selective electrodes (ISEs) bridges fundamental host-guest chemistry, membrane science and its specific applications. Because of their simplicity,low cost,sufficiently reliable and repeatable measurements ISEs are recognized as novel analytical tools for selective determination of analyte ions[1,2].

Many Na-ISE incorporating a various ion carriers have been reported.In the present study,a simple heterogeneous precipitate based membrane have been prepared. Along with the potentiometric performances of these sensors, effect of p^H, effect of medium, response time and selectivity coefficients with respect to different interfering ions have also been studied.

Experimental

Preparation of Heterogeneous precipitate:

To 10 ml of aqueous NaOH (Finer reagents, extra pure) with normality of 0.9246 was mixed with 3ml of methyl salicylate (Burgoyne, urbidges & Co India). It is heated for 1hour 20minutes. After heating the solution was cooled for 1-2 hours and acidified with conc. HCl. A white precipitate was obtained. It was filtered and dried in air for 12hours and powdered.

Preparation of methyl salicylate based membrane electrode :

About 0.2g of powdered precipitate was thoroughly mixed with Epoxy resin (Huntsman advanced materials, India Pvt. Ltd) and the paste was applied on Whatmann filter paper No.42. This was spread uniformly over the filter paper to obtain 0.9mm thickness of the electro active materials with matrix. This was left in air to dry for 48hours to get an electroactive membrane. A circular piece of this membrane was cut and fixed with resin a one end of the hollow glass tube (diamete 0.6cm and length 6cm). This tube was fille with saturated solution of $CuSO_4$ and referenc copper metal wire was inserted (diamete 0.5mm & length 12cm) through other end c $CuSO_4$ solution already filled in this glass tub This complete assembly will work as an io selective electrode for sodium ion determ- nation. This ion selective electrode was ke in 1M solution of sodium chloride for one we to attain equilibrium.

The entire electrode system for t measurement can be represented as

Internal reference Electrode	Internal reference solution (saturated $CuSO_4$ solution)	Ion selective membrane	Sample solution	External referenc electrode (Ag-A electrode)

Results and Discussion

Response Characteristics of the electrode:

The Electrode was first conditioned in 1M solution of sodium ion till it attained stable equilibrium after which it was used for the determination of characteristic study of the electrode. The electrode potential for a series of standard solution of Na(I) ions was measured. The electrode gave a linear response to Na(I) concentration in the range of 1M to 1×10 The sodium-ISE reveals near Nernstian s 25mv/decade graph-1.

To find the response time the ele was first dipped in 1M of Na(I) solutio suddenly the concentration of the soluti changed to 0.1M. The variation in po was noted at every 5 seconds till a co potential was obtained at 4minutes and constant.

Table 1. Electrode Response

Concentration of sodium chloridesolution (M)	E.M.F (in volts)
1	-0.023
1×10^{-1}	-0.016
1×10^{-2}	-0.007
1×10^{-3}	-0.004
1×10^{-4}	-0.002
1×10^{-5}	-0.001
1×10^{-6}	-0.001

Effect of pH and Effect of medium:

To study the effect of pH, a standard solution containing 1M sodium ion were prepared in which a series of buffer solution 4.01 to 9.2 was added. It was found that the potential remained unchanged with in the pH range 4.01-9.2.

To study the effect medium, a standard solution containing 1M Na(I) ion in a series of 25%,50%,75% ethanol was added. It was found that the potential remains unaffected in ethanol medium.

Selectivity:

The selectivity, which is important characteristics of a membrane sensor, is measured in terms of potentiometric selectivity coefficient $K^{pot}_{Na,M}$. It measures the response of the sensor towards the primary ion in the presence of secondary ion present in the sample solution. The selectivity[2-5] coefficient has been determined by using fixed interference method (FIM) based on semi empirical Nicolski-Eisenman equation. In this method the concentration of primary ion Na (I) ion is varied, where as the concentration of secondary interfering ion is kept constant in the sample solution which is 1×10^{-1} M concentration of interfering ion in the present case. The potentiometric selectivity data of sensors for various interfering ions is given in table 2 & 3. These ions don't disturb the normal functioning of the sensors and the sensors possess high sodium selectivity and respond weakly to these interfering ions

Table 2. Interference by Cations

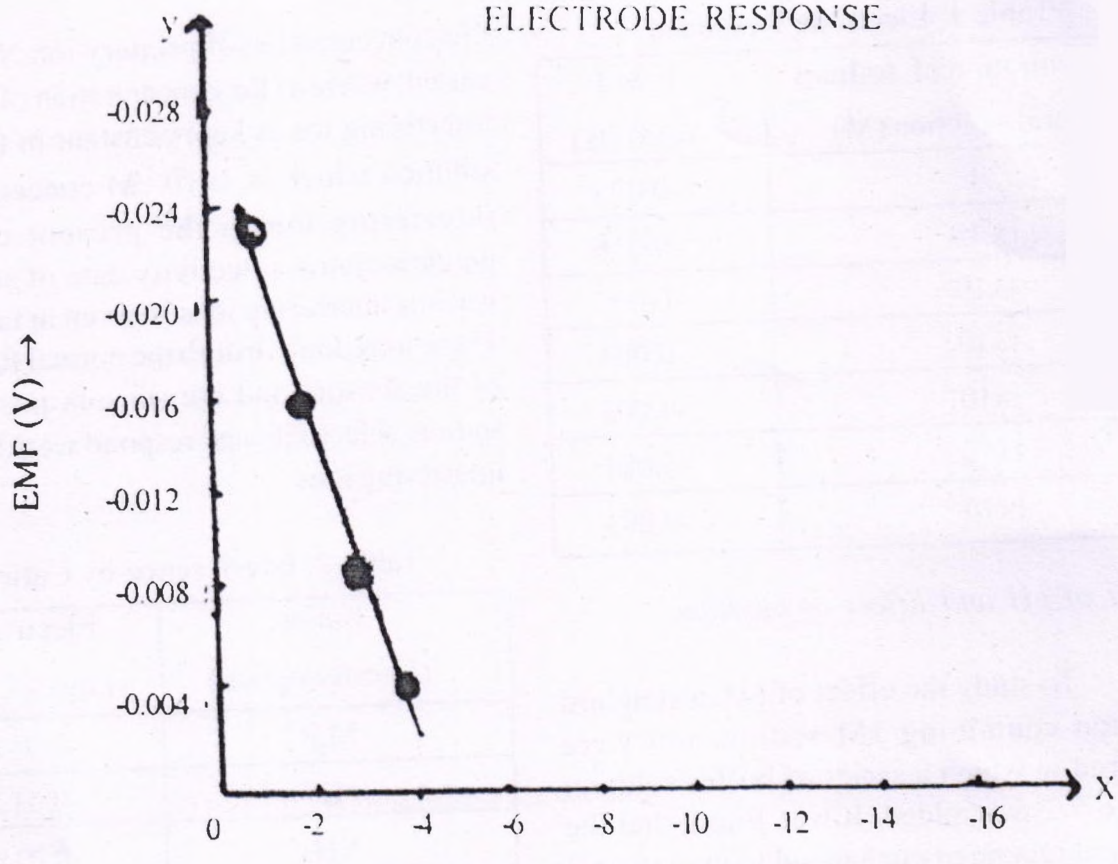
Cation (Interfering ion)	Electrode Log $K^{pot}_{Na,M}$
Mg^{2+}	-0.018
K^{+}	-0.017
NH_4^{+}	-0.018
H^{+}	-0.018

Table 3. Interference by Anions

Anions (Interfering ion)	Electrode Log $K^{pot}_{Na,M}$
SO_4^{2-}	-0.018
I	-0.017
Cl^{-}	-0.017
Br^{-}	-0.018
$S_2O_3^{2-}$	-0.017
$C_2O_4^{2-}$	-0.018
urea	-0.018

Analytical applications :

To assess the applicability of the sensor to real samples, an attempt was made



Plot of E.M.F. Vs log concentration of Sodium (M)

to determine sodium ion in real samples like commercial baking powder and ala. The recovery of sodium ion in sample analysis was found to be quantitative with the maximum recovery of 95%.

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