A Heterogeneous Precipitate Based Sodium Membrane Ion Selective Electrode-Its Preparation and Analytical Application

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ABSTRACT

A heterogeneous precipitates have been as ion carriers for the preparation of Na (I) selective membrane sensor. This electrode give near–nernstian responses in linear concentration range of 1M to 1×10^{-4}M with detection limits of the order of 10^{-4}M . The stable potentiometric signals are obtained with in a short time period of 3 minutes. The effect of pH, and the effect of medium have been studied found to give a better response. Selectively coefficient values (log $K^{pot}_{Na,M}$) have been evaluated using fixed interference method. The sensors have also been used as indicator electrode in commercially available products.

Key words: A heterogeneous peecipitate, 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, textile ,drinking water, soaps detergents and a as a drain cleaner.

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

The field of Ion–selective electrodes (ISEs) bridge fundamental host guest chemistry, membrane science and its specific application. Because of their simplicity, low cost, sufficiently reliable and respectable measurement, 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 sensor, effect of pH, effect of medium, response time and selectivity coefficients with respect to different interfering ions have also been studied.
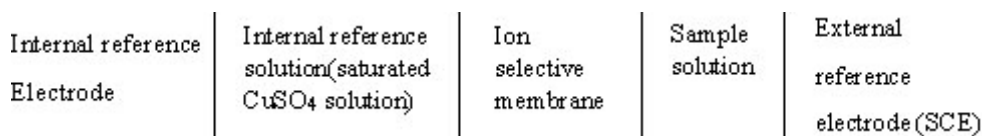
EXPERIMENTAL

Preparation of S-benzyl isothiouronium chloride based membrane electrode

About 0.5g of benzoic acid (HI-PURE,FINE CHEM INDUSTRIES) was to 6 ml of aqueous NaOH (FINAR REAGENTS, Extra pure) of strength 0.9246 N. Mixed the resulting solution with an equal volume of S-Benzyl isothiouronium chloride reagent (HIMEDIA Labaratories PVT.Ltd).A white precipitate was obtained. It was filtered and dried in air for 12 hours and powdered. About 0.2g of powdered precipitate was thoroughly mixed with Epoxy resin (Huntsman advanced materials) and the paste obtained 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 48 hours to get an electro active material 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 at one end of the hollow glass tube (diameter 0.6cm and length 9cm). This tube was filled with saturated solution of $CuSO_4$ and reference copper metal wire was inserted (diameter 0.5mm & length 12cm) through other end of $CuSO_4$ solution already filled in this glass tube. This complete assembly will work as an ion selective electrode for sodium ion determination. This ion selective electrode was kept in 1M solution of sodium chloride for one week to attain equilibrium.

The entire electrode system for the measurement can be represented as

Internal reference Electrode	Internal reference solution(saturated $CuSO_4$ solution)	Ion selective membrane	Sample solution	External reference electrode(SCE)

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) ion was measured. The electrode gives linear response to Na(I) ion concentration in the range of 1.0 M to 1×10^{-4}M (Table 1). The sodium - ISE revealed near nernstian slopes 25 mV/decade. Graph-1
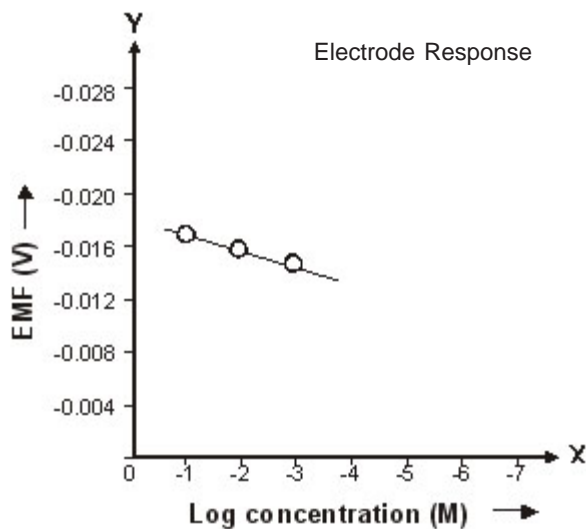
To find the response time, the electrode was first dopped in 1M of Na (I) solution and suddenly the concentration of solution was changed

Table 2: Interference by cations

Cation (Interfering ion)	Electrode Log $K^{pot}_{Na,M}$
Mg^{2+}	-0.030
K^+	-0.029
NH_4	-0.030
H^+	-0.028

Table 3: Interference by anions

Anions(Interfering ion)	Electrode Log $K^{pot}_{Na,M}$
SO_4^{2-}	-0.030
I^-	-0.029
Cl^-	-0.029
Br^-	-0.030
$S_2O_3^{2-}$	-0.031
Urea	-0.028
$C_2O_4^{2-}$	-0.028

Table 1: Electrode response

Concentration of Sodium Chloride solution(M)	E.M.F (volts)
1	-0.029
1×10^{-1}	-0.027
1×10^{-2}	-0.017
1×10^{-3}	-0.007
1×10^{-4}	-0.003

Graph 1: Plot 8 EMF vs log concentration of sodium (M)

to 0.1 M. The variation in potential was noted at every 5 seconds till a constant potential value obtained at 3 seconds and remains constant

Effect of pH

To study the effect of pH, a standard solution containing 1M sodium ion were prepared in which a series of buffer solution 4.02 to 9.2 was added. It was found that the potential remains unchanged with in the pH range of 4.01-9.2.

To study the effect of 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 an important characteristics of a membrane sensor is measured in terms of the potentiometric selectivity K, it measures the response of the sensor towards the primary ion in the presence of secondary ion present

in the sample solution. The selectivity coefficient has been determined by using fixed interference method (FIM) based on semi empirical Nicolskii-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 $1 \times 10^{-1}M$ concentration in the present case. The potentiometric selectivity coefficient data of sensors for various interfering ions given in Table -2&3. These ions don't disturb the normal functioning of the sensors and the sensors posses high sodium selectivity and respond weekly to these interfering ions.

Analytical application

To asses the applicability of the sensors to real samples an attempt was made to determine sodium ion in real samples like commercially available baking powder and ala. The recovery of sodium ion in sample analysis was formed to be quantitative with the maximum recovery of 95%.

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