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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. The electrodes give near–nernstian responses in linear concentration range of 1M to $1x10^{-2}$M with detection limits of the order of 10^{-2}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 as an indicator electrode in commercially available products.

Key words: A heterogeneous peecipitate, Na (I) ion, membrane sensor, Fixed interference method.

ntroduction

Sodium hydroxide is a caustic metallic ase. It is used in many industries, mostly as a rong chemical base in the manufacture of pulb nd paper, textile drinking water, soaps detergents nd a as a drain cleaner.

Food uses of sodium hydroxide include shing or chemical peelingof fruits and vegetables hocolate and cocoa processing caramel colouring roduction, 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 Process

I) Preparation of Heterogenious 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 and acidified with conc.HCl.A white precipitate was obtained. It was filtered and dried in air for 12hours and powdered.

II)Preparation of methyl salicylate based membrane electrode using PVC and DOP :

About 0.1g of dried precipitate was thoroughly mixed with 0.1g of polyvinal chloride (KEMPHASOL Bombay) and adde Dioctyl Phthalate plasticier,(LOBAL CHEM laboratory Reagents & Fine Chemicals) T paste obtained was applied on whatmann filt paper no.42. This was spread uniformly ov the filter paper to obtain 0.9mm thickness the electro active materials with matrix. Th was left in air to dry for 24 hours to get an elect active membrane. A circular piece of th membrane was cut and fixed with resin at on end of the hollow glass tube (diameter 0.6cm and length 6cm). This tube was filled wi saturated solution of $CuSO_4$ and reference copp metal wire was inserted (diameter 0.5mm length 12cm) through other end of CuS solution already filled in this glass tube. Th complete assembly will work as an ion select electrode for sodium ion determination. Th ion selective electrode was kept in 1M solut of sodium chloride for one week to att equilibrium.

The entire electrode system for the measurem can be represented as

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

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 seri standard solution of Na(I) ion was measu The electrode gives linear response to Na(I concentration in the range of 1.0M to 1x10 (Table 1). The sodium - ISE revealed n nernstian slopes 27mV/decade. Graph-1

To find the response time, the elect

was first dopped in 1M of Na (I) solution and suddenly the concentration of solution was changed 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

Table 1. Electrode response

Concentration of sodium Chloride solution (M)	E.M.F volts
1	-0.019
$1x\ 10^{-1}$	-0.010
$1x10^{-2}$	-0.005

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 1.02 to 9.2 was added. It was found that the potential remained unchanged with in the pH range 4.01-7.

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 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 $1x10^{-1}$ 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.

Table 2. Interference by cations

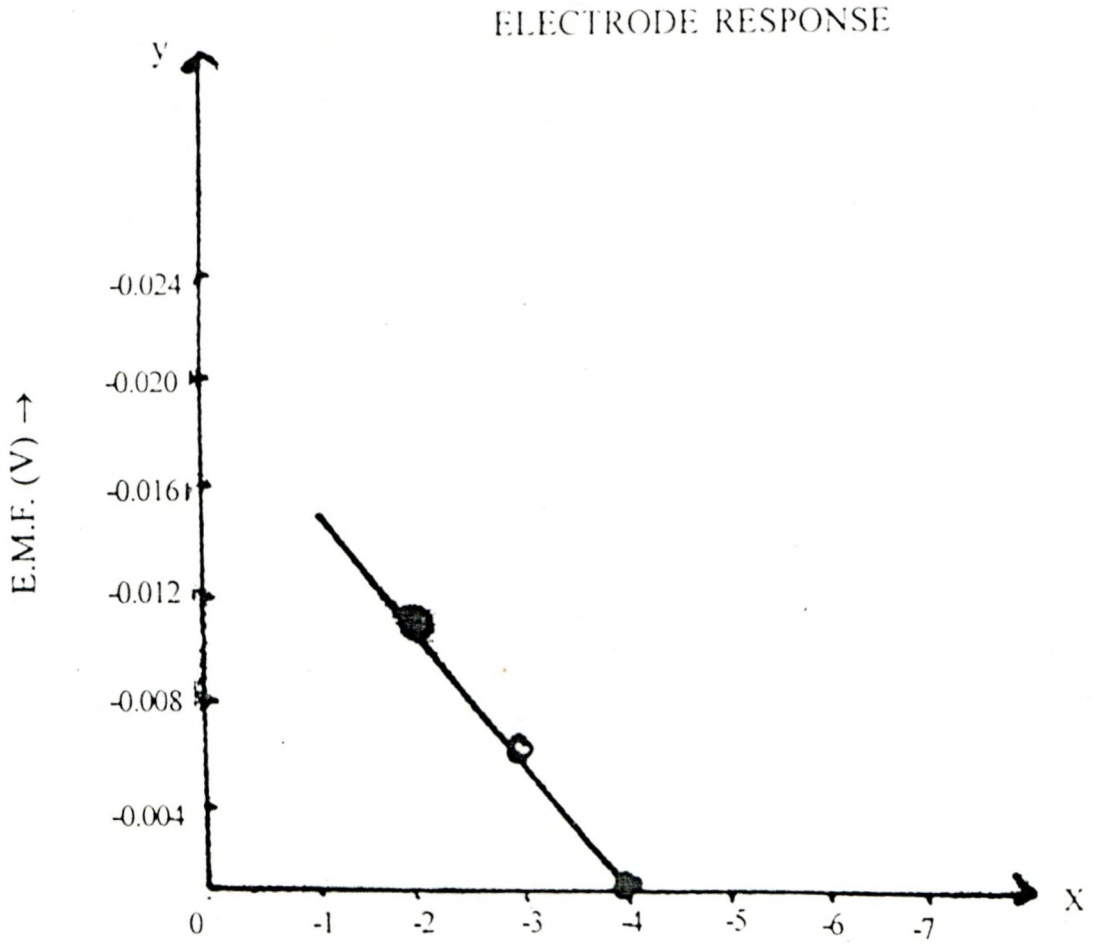
Cation (Interfering ion)	Electrode $\log K^{pot}_{Na,M}$
Mg^{2+}	-0.030
K^{+}	-0.029
NH_4	-0.029
Na^{+}	-0.030
H^{+}	-0.028

Table 3. Interference by anions

Anions (Interfering ion)	Electrode ($\log K^{pot}_{Na,M}$)
SO_4^{2-}	-0.030
I^{-}	-0.029
Cl^{-}	-0.028
Br^{-}	-0.029
$S_2O_3^{2-}$	-0.030
NH_2-CO-NH^{-}	-0.028
$C_2O_4^{2-}$	-0.029

Analytical application:

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



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

recovery of 95%.

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