

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 1×10^{-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 ($\log K_{Na,M}^{pot}$) 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.

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 cleaning 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 Process

I) Preparation of Heterogenous Precipitate:

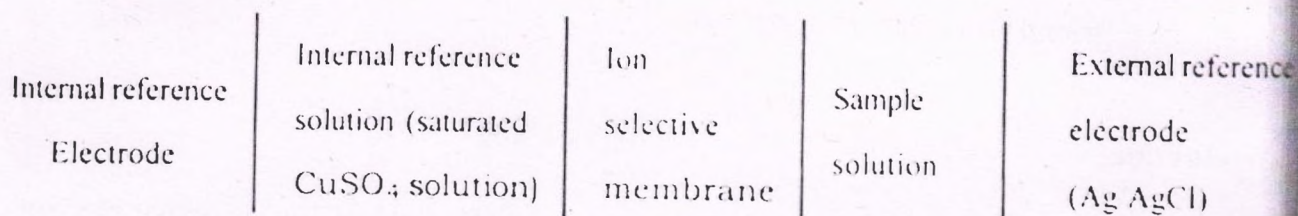
To 10 ml of aqueous NaOH (Finer reagents, extra pure) with normality of 0.9246 was mixed with 3ml of methyl salicylate (Burgoyne, urbidges & Co India). It is heated for 1 hour 20 minutes. After heating the solution was cooled and acidified with conc. HCl. A white precipitate was obtained. It was filtered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

chloride (KEMPHASOL Bombay) and added Dioctyl Phthalate plasticizer (LOBAL CHEMICALS laboratory Reagents & Fine Chemicals). 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 24 hours to get an electro active membrane. A circular piece of the membrane was cut and fixed with resin at one end of the hollow glass tube (diameter 0.6cm and length 6cm). This tube was filled with saturated solution of CuSO_4 and reference copper metal wire was inserted (diameter 0.5mm and length 12cm) through other end of CuSO_4 solution already filled in this glass tube. The complete assembly will work as an ion selective electrode for sodium ion determination. The 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



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 standard solution of Na(I) ion was measured. The electrode gives linear response to Na(I) concentration in the range of 1.0M to 1×10^{-5} (Table 1). The sodium - ISE revealed a Nernstian slope of 27mV/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 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 |
| 1×10^{-1} | -0.010 |
| 1×10^{-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 0.2 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) is varied where as the concentration of

secondary interfering ion is kept constant in the sample solution which is 1×10^{-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.

Table 2. Interference by cations

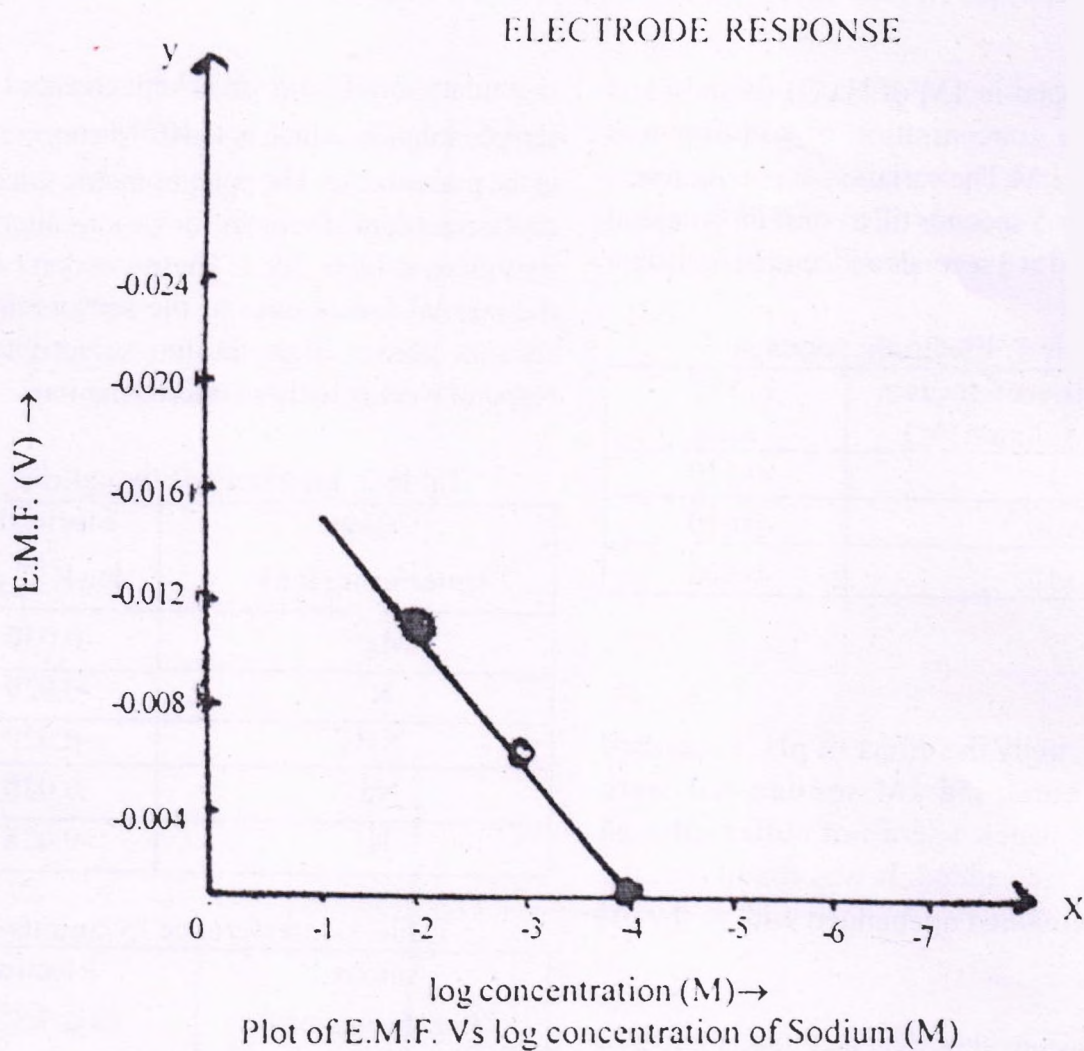
| Cation (Interfering ion) | Electrode $\log K_{Na,M}^{pot}$ |
|--------------------------|---------------------------------|
| Mg^{2+} | -0.030 |
| K^+ | -0.029 |
| NH_4 | -0.029 |
| Na^+ | -0.030 |
| H^+ | -0.028 |

Table 3. Interference by anions

| Anions (Interfering ion) | Electrode $(\log K_{Na,M}^{pot})$ |
|--------------------------|-----------------------------------|
| 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



recovery of 95%.

References

1. Bakker E, Bahlmann P & Pretsch E, *Chem. Rev.* 97 (1997) 30383 (b) Buhlmann P Pretsch E and Bakker E, *chem. Rev.* 98, 1593 (1998).
2. Chandra S., Agarwal H., Singh C.K., Sindhu S.K. and Kumar P., *Ind. J. Chem.* 44A (2005) 2060; (6) Nanda D, Chouhan HDS Maiti B, *Ind. J. Chem.* 43(A) 1685 (2004).
3. Asila Al-Musheifri and Graham R.D. Johns "Glucose interference in direct ion-selective electrode sodium measurement", *Anna clinical Biochemistry*, 45, 530-532 (2006).
4. Bijster and Vink, "Direct potentiometric determination of sodium ion in blood potentiometric response in simple sodium chloride solutions", *clinical chemistry*, 6-10 (1984).
5. Chapman and Goldsmith, "Determination of chloride, sodium and potassium in food stuffs using ion-selective electrodes the dry sample addition method," *Analyst*, 1014-1018 (1982).