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A NEW SELECTIVE ELECTRODE FOR ANALYSIS Nickel (II) IONS BY USING CARBON PASTE ELECTRODE (CPE) $_{\text{Nickel}}$ MODIFIED

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ABSTRACT

In that work , We Adopt Preparation anew Carbon Paste electrode for Nickel ions based on complex [3-(4-nitro phenyl azo)-pentane-2,4-dion]:(**LP**) With Nickel Ions As electroactive material The best component for Carbon paste electrode – Nickel (II) ions :(7% Ionophore ,35,50% Graphite powder ,46.50% Plasticizers (Di Butyl phthalate) ,7 % NaTBP, 3% Nio, 1% Graphene oxide)**.**The electrode exhibited a Nernstian slope of (29.30±1) mv per decade of Nickel Ions over concentration range of ($0.75^{x}10^{-7}$-$0.50^{x}10^{-1}$) M in The PH range (4.00 – 10.00).By using (MPM) Matched Potential Method This electrode revealed good Selectivity for Nickel Ions over wide variety of other metal ions with (R= 0.999).The detection limit was (3.5^{x} 10^{-8}) M and The response time was about (15) S. The Electrode can be used for at least 3 months without a considerable divergence in potential response. the proposed electrode was used for determination of Nickel in a standard solution and was successfully applied as an indicator electrode for potentiometric titration of Nickel Ions with EDTA.

INTRODUCTION

Nickel derives its name from the Swedish language and the word (Kopparrnickel) meaning unreal copper.Isolated by the Swedish scientist Axel Clonsted in 1705. Nickel is the twenty-second most abundant element in the Earth's Crust.Nickel is an essential minor component for humans, maximun permissible limit in drinking Water (**0.040-0.070**)mg/l.[Mohammed, Gharam I; et al]. Nickel contributesto the effective production of green hydrogen using the hydrogen fuel cell as one of the environmentally friendly forms and production of alternative renewable energy[Maallah, Raja; et al]. It is used in the manufacture of metal alloys, mining, electroplating, oil refining catalysts, and electrochemical fuel cells that convert chemical reactions into electrical energy as a good candidate to fill the gap.advanced batteries for powering portable devices.

Nickel production is increasing every year due to the rapid improvement in industry rates. Metal polution has recived impressive attention, Making it a source of concern for environmentalists as it poses health risks of pneumonia, skin, asthma, lung Cancer, and is responsible for kindly disorder and blood disorders. As a result of these dangerous effects, it is necessary to selectively monitor nickel ions in industries and biological and environmental systems[Verma, Manish; et al].

The Current techniques existed in the Litreature for determain of Nikel(II) include : Inductivity coupled plasma – atomic emission spectroscopy (ICP-AES) [Kumar, M Kiran; et al],Inductivity coupled plasma- Mass

spectroscopy (ICP-MS) [Russel, BC; et al *.Retka, j;et al],Flame atomic absorption spectrometry [Khudhair, Ahmed Fadhil; et al],X-ray Flourecence (XRF) [Hasan, M.S;et al *.] , High Performance Liquid Chromatography [Chen, Fang;et al], Stripping Voltametry [Nur Abdul Aziz, Siti Fathimah; et al] ,and Neutron Activation analysis (NAA) [Chae, San; et al] Are various instrumental methods used for determination Nickel (II) ions[Mossotti, Giulia;et al] .However, These methods are time consuming,multi steps and require large infrastructure and are too expensive for many analytical laboratories .Thus ,the development of covenient and direct method to assay of Nickel ions in different samples is urgently needed .Among the various classes of chemical sensors , ion-Selective electrodes were described (1906) and offered great advantages such as wide linear working range,low detection limits ,low-energy consumption,short response time hight selectivity and sensevity, lowcost [Alizadeh, Kamal;et al] precisely , Carbon paste modified electrodes have appeared as a model of modified models for ion selective electrodes [Adams R,] ,

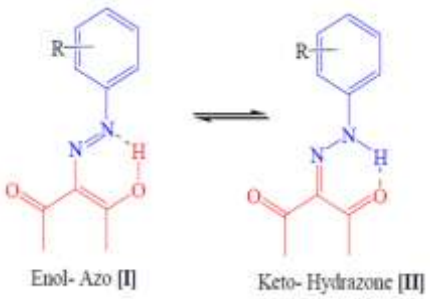
The ion selective Carbon paste electrode (ISCPE) and potentiometry method have some advantages over other membrane electrodes: such as "renewability , stable response, low ohmic resistance ,no need for internal solution, [KUWANA, T; et al]. They are the most frequent use of Potantiometric sensors in analytical chemistry also in biochemical and biophysical fields.

In this paper ,we aim to provide an overview of the current advancees in the Nickel (II) – Selective potentiometric electrodes and its applications.

EXPERIMENTAL DETAILS

2.1. Reagent and materials:

Azo pigment [3-(4-nitro phenyl azo)-pentane-2,4-dion] :(**LP**) :(Dye) was purchased from MERCK. The graphite powder (G) and Graphene Oxide (GO) (MERCK) with the paraffin oil, Di Octyl Sebcate, Di Butyl Phthalate, Di Octyl Phthalate (Aldrich) was high purity, Nickel (II) Nitrate GR pro analysis and Nitrate salts of cations were purchased from MERCK. Deionized water was used for preparing all of the Solutions.



(Azo Pigment)

2.2. Construction of Carbon Paste Selective Ni (II) electrode– (CPE-Ni (II)Lp-Azo):

Carbon paste electrode preparation desired of 7% Azo pigment, 35.50% graphite powder, 46.50% Di Butyl phthalate oil, 3% Nickel oxide, 1% Graphene Oxide and 7% tetra phenyl borate sodium (**TPBNa**): as material to good Response Nernstian, these components were thoroughly mixed and then transferred into (syringe, glass tube), of i.d.5 mm and a high of 3cm [Habibi, N; et al]. After the mixture was uniformed as well as possible, to avoid the formation of air gaps, the paste was packed carefully into the syringe (tube). This can prevent the increase of the resistance of the electrode. Then the electrical contact was made by inserting silver wire in to the opposite end. The working surface of the electrode was polished by using soft abrasive paper, then; the electrode was conditioned for 24 **h** by being soaked in a $(1.0^{x}\ 10^{-2})$ mol. l^{-1} of Ni $(No3)_2.3H2o$ solution. Then the electrode was used for potentiometric measurements of this metal cation in solution. **Figure (1).**

The type and the amount of the Carbon paste electrode **(CPE)-Ni** $_{(II)}$ ingredients ionophore, graphite powder, Plasticizers, etc) are important factors affecting the selectivity and stability of the ion selective electrode [Ejhieh, Nezamzadeh.A.]. therefore, it is essential to optimize their values to construct a selective

electrode [Sighl, p; et al *. Isildak, Omer.] with suitable electrochemical characteristics.

In order to choose a suitable plasticizer, several tests were performed using PARAFFIN**OIL, Di Octyl Sebcate (DOS), Di Octyl Phthalate (DOP)** and **Di Butyl phthalate oil (DBP**). the experimental results showed that a good Nernstian behaviour is observed in the presence of Di Butyl phthalate oil. Therefore, we use this plasticizer in in the construction of the proposed electrode. The ranges of the amounts of the ingredients of the selective (carbon paste electrode) Nickel which were studied shown in table (1).

Figure (1): ingredients and preparing Carbon Paste Selective Ni (II) electrode

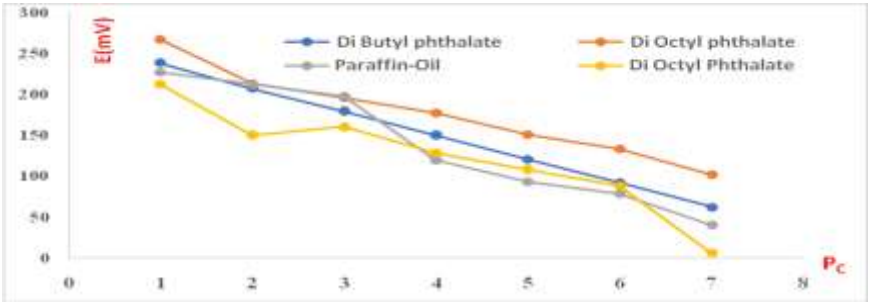
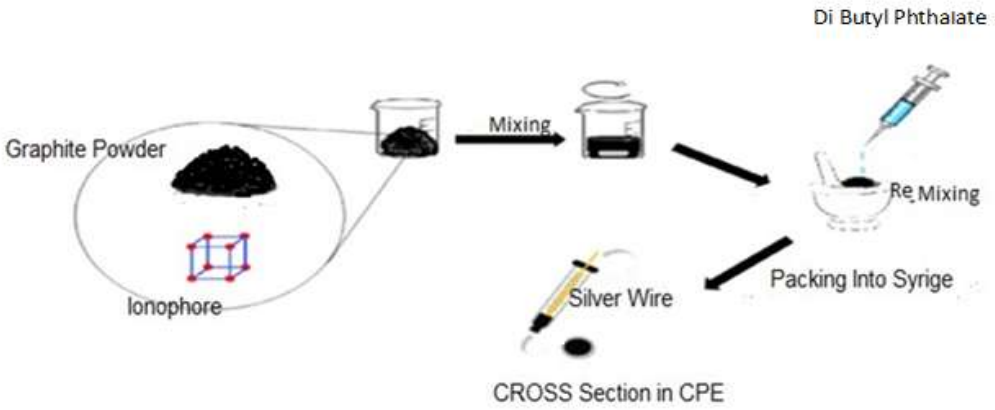


Figure (2): Effect of plasticizes on Carbon Paste Selective Ni (II) electrode– (CPE-Ni (II)Lp-Azo)

Table (1): The best component for Carbon paste electrode – Nickel (II) ion					
Ionophore%	**Graphite Powder %**	**Go%**	**DBP%**	**NaTBP%**	**Nio%**
7%	**36.50**	**1%**	**46.50%**	**7%**	**3%**

2.3. Apparatus and Potentiometric measurements:



All potential measurement were carried out by the Following the electrochemical cell, where the electrochemical cell contained: a silver – silver chloride double – junction reference electrode REF361 (one 274-F01) as a reference electrode and the Nickel (II) sensor as an indicator electrode [Hasan, Ali Kamel; et al]. The schematic figure (3) is as follows:

Ag- Agcl (Saturated) ‖ Sample Solution ‖Carbon paste electrode.

After **EMF** is Calculated, Calibration curve is drawn by plotting potential, versus, the logarithm of the Nickel ions concentration**s.** We notice increase E(mv) for cell with increase the Nickel ions concentration

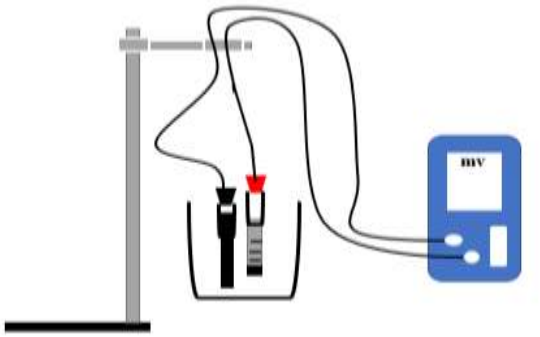


Figure (3): electrochemical Cell system by using (CPE- nickel) proposed as an indicator electrode and Ag-Agcl as a reference electrode.

3.0. RESULTS AND DISCUSSION

3.1. Influence of nature and polarity of plasticizers on electrochemical response of Carbon Paste Selective Ni (II) electrode - (CPE-Ni (II)Lp-Azo):

The response of an ion selective electrode depends on the amount and the nature of the ingredients of the carbon paste electrode.plastizers plays an important role in performance ion-selective electrodes[Aglan RF, et al], and the nature of plasticizers influences the dielectric constant of the carbon paste phase ,the mobility of the ionophore molecules in the carbon paste and also the state of ionophore [Faridbod F, et al], therefore, we preferred to choose the type of plasticizers .thus , several carbon paste electrode with similar composition of graphite powder ,ionophore and additive ,but four different composition of plasticizers with different dielectric constant were prepared and tested.

The result and summarized in table (2). The best electrochemical response was obtained in presence of (Di Butyl Phthalate oil:DBP). Since the carbon, paste has a high lipophilicity, high molecular weight, low vapor pressure, and low tendency for exudation from the polymer matrix and high capacity to dissolve the substrate and the other additives present in carbon paste and also they should possess an adequate dielectric constant.DBP oil with high dielectric constant and good polarity can be affected the characteristics of the carbon paste and ionophore. **Figure (2).**

3.2. Determination of optimum effects of Carbon Paste Selective Ni (II) electrode - (CPE-Ni (II)Lp-Azo) ingredients:

In the next step, for determination the optimum amount of carbon paste electrode ingredients. Ionophore, graphite powder, plasticizers and additive were chosen as the variables towards the electrochemical response of the constructed electrode .the amount of. Ionophore, graphite powder, plasticizers and additive [Jingni H; et al] that were selected and listed in table (3) .these components represent 100 weight-percent of the total formulation that is:

A+B+C+D+E+F = 100%

A: Ion-exchanger
B: Graphite
C: Plasticizer
D: NaTBPH
E: NiCkel Oxide
F: Graphene oxide

Table (2): Effect polarity of plasticizers on the Nernstian slope			
Paraffin	DOPH	DOS	DBPH
22.22 mv/drecade	26.80 mv/drecade	24.20 mv/drecade	29.30 mv/drecade

3.3. The pH dependence of Carbon Paste Selective Ni (II) electrode - (pH CPE-Ni (II)Lp-Azo):

The pH dependence of the electrode response was determined by using a solution with pH ranging from 1to 13. The pH was adjusted with hydrochloric acid and Sodium hydroxide. The Carbon Paste Selective Ni (II) electrode showed a Potential change against the Nickel Concentrations of (1.0×10^{-1} -1.0×10^{-2}-1.0×10^{-3}) mol. l^{-1} in acidic and basic mediums. The protentional remained unaffected over a pH range (4.0-10.0)**.** the change in potential at low pH values, may be attributed to the response of the electrode to the H_3O^+ ions I solution . At higher pH values than 10.0, the Ni(II) ions may interact with OH^- ions in solution [Alizadeh, Taher et al*. Samizadeh, Mehdiet al]. Figure (4).

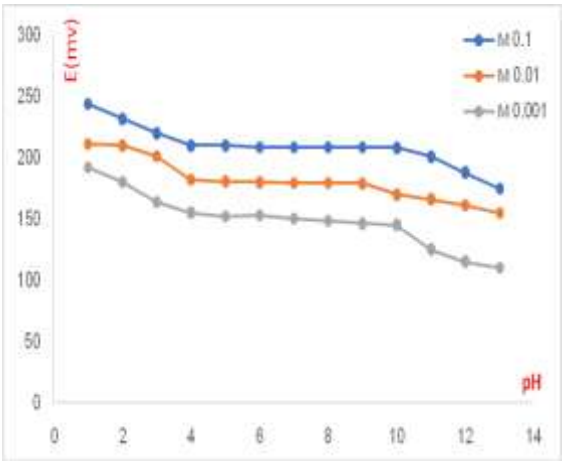


Figure (4): effect of pH on potential response Carbon Paste Selective Ni (II) electrode– (CPE-Ni (II)Lp-Azo)

Table (3): The precentage of composition ofCarbon Paste Selective Ni (II) electrode - (CPE-Ni (II)Lp-Azo):

G/P	R^2	Linear range	mv/decade	F	E	D	C	B	A	
1.0	0.896	10^{-5}-10^{-3}	19.17	**0%**	**0%**	**0%**	48.50%	48.50%	**3%**	1
1.0	0.999	10^{-5}-10^{-2}	21.40	**0%**	**0%**	**0%**	47.50%	47.50%	**5%**	2
1.0	0.998	10^{-4}-10^{-2}	23.37	**0%**	**0%**	**0%**	46.50%	46.50%	**7%**	3
1.0	0.998	10^{-5}-10^{-1}	21.00	**0%**	**1%**	**0%**	6.50%4	45.50%	**7%**	4
1.0	0.789	10^{-6}-10^{-1}	23.29	**0%**	**2%**	**0%**	46.50%	44.50%	**7%**	5
1.0	0.897	10^{-5}-10^{-1}	24.18	**0%**	**3%**	**0%**	46.50%	43.50%	**7%**	6
1.0	0.988	10^{-7}-10^{-1}	20.22	**0%**	**3%**	**1%**	46.50%	42.50%	**7%**	7
1.0	0.999	10^{-7}-10^{-1}	25.02	**0%**	**3%**	**3%**	46.50%	40.50%	**7%**	8
1.0	0.988	10^{-5}-10^{-1}	25.28	**0%**	**3%**	**5%**	46.50%	38.50%	**7%**	9
1.0	0.898	10^{-4}-10^{-1}	26.01	**0%**	**3%**	**7%**	46.50%	36.50%	**7%**	10
1.0	0.999	10^{-7}-10^{-1}	29.30	**1%**	**3%**	**7%**	46.50%	35.50%	**7%**	11

3.4. Response time of Carbon Paste Selective Ni (II) electrode- (CPE-Ni (II)Lp-Azo):

The response time of the carbon paste electrode is an important parameter for their analytical application. After successive immersion of the electrode in a series of its respective ion solutions, each concentration has 10-fold increase than in concentration previous (1.0×10^{-6}M to 1.0×10^{-1}M) of Ni (II) ions solutions. (Darroudi, Abolfazl*. El said, Fathi Aet al) The response of the electrode is stabilized after (15) S compared with before concentration, as is shown in figure (5).

3.5. Life time of Carbon Paste Selective Ni (II) electrode- (CPE-Ni (II)Lp-Azo): The lifetime of an electrochemical sensor based on carbon

paste electrode matrix, depended on the distribution coefficient of the ionophore and plasticizer between carbon paste electrode and aqueous phases and also depended on how to use the electrode [Shamra, Harish K; et al]. the experimental results were shown that the constructed could be used for at least (15) weeks without any change in the Nernstian slope and detection limit Figure (6).

3.6. Analytical applications of CPE-Ni (II)$_{Lp\text{-}Azo}$: The fabricated **CPE-Ni (II)** was used as an indicator electrode in successful titration of 25 ml of Ni (II) ions (2^x 10^{-4}M) with standard solution of EDTA (1^x10^{-2} M) at pH (7.0) The resulting titration curve for titration of Ni (II) ions solution with the solution of EDTA, which is presented in figure (7), shows that amount of Ni (II) ions in solution can be accurately determined with the fabricated selected electrode**.**

3.7. Selectivity Coefficient of CPE-Ni (II)$_{Lp\text{-}Azo}$: Selectivity is one of the most important characteristics of electrode, which defines the extent to which it may be employed in the determination of a particular ion in the presence of other interfering ions. Potentiometric selectivity coefficients of the Carbon paste Nickel (II) electrode: **CPE-Ni (II)$_{Lp\text{-}Azo}$** were evaluated by matched potential method (MPM), ∂ specified activity (concentration) of primary ions is added to a reference solution, and the potential is measured. In other experiment, interfering ions(B) are added to an identical reference solution until the measured potential matches that obtained before the addition of primary ions. In this method selectivity coefficient**,** $K_{A,B}^{MPM}$ is then given by the resulting primary ion to interfering ion activity (concentration)ratio:

$$K_{Ni^{+2},M^{+n}}^{MPM}=\frac{(a_A{}'-a_A)}{a_B}$$

∂_A is primary ion activity (concentration) and ∂_B is interfering ion activity (concentration) [Larif, Fekri; et al*. Ardakani, Mazloum M; et al]. the resulting selectivity coefficients of: **CPE-Ni (II)$_{Lp\text{-}Azo}$** in the presence of various interfering ions is in the order of (1^x10^{-2}) M or less. As a result, there is a good performance of the fabricated: **CPE-Nickel (II)$_{Lp\text{-}Azo}$**towards the Ni (II) and interaction between Ni (II) Ions and complex is effectivity

The results are given in the table (4). The electrode has a reasonably good selectivity over some common alkali and transition ions. Selectivity coefficient values are better at lower interfering ion concentration**.**

Table (4): Selectivity Coefficients ($K_{Ni^{+2},M^{+n}}^{MPM}$) of various interfering metal ions

Interfering ion	∂_B	$K_{Ni^{+2},M^{+n}}^{MPM}$	**Interfering ion**	∂_B	$K_{Ni^{+2},M^{+n}}^{MPM}$
Ca^{2+}	1.726×10^{-3}	2.838^x10^{-4}	Cr^{3+}	3.381×10^{-4}	1.449^x10^{-3}
Mg^{2+}	2.200×10^{-4}	2.227×10^{-3}	Cd^{2+}	2.630×10^{-3}	1.863^x10^{-3}
Fe^{3+}	2.480×10^{-3}	1.975×10^{-4}	Al^{3+}	3.163×10^{-4}	1.549^x10^{-3}
Co^{2+}	2.880×10^{-3}	1.701^x10^{-4}	Cu^{2+}	2.176×10^{-3}	2.251^x10^{-4}
Pb^{2+}	3.100×10^{-3}	1.580^x10^{-4}	As^{+1}	2.969×10^{-4}	1.650^x10^{-3}
Ag^{+1}	2.668×10^{-4}	1.836×10^{-3}	Zn^{2+}	1.931×10^{-4}	2.530×10^{-3}

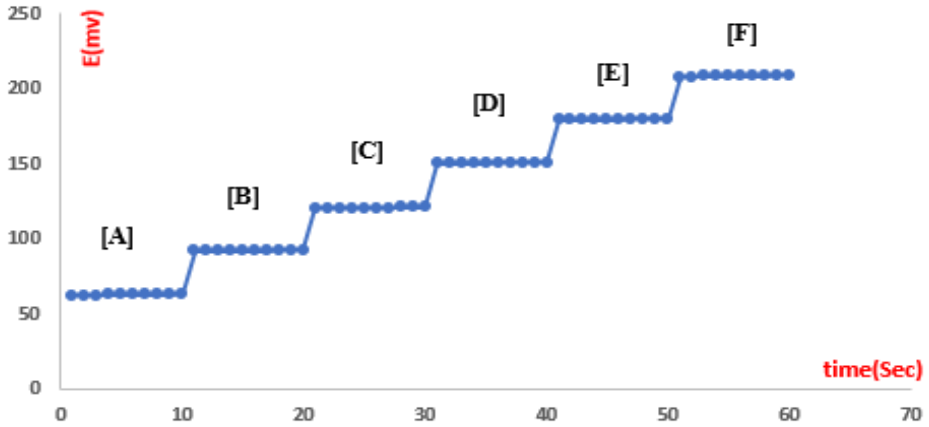


Figure (5): Static potential –Time plots for different concentrations of Nickel (II) ions (A)$1.0^{x}10^{-6}$M, (B)1.0^{x} 10^{-5} M, (C)$1.0^{x}10^{-4}$ M, (D)$1.0^{x}10^{-3}$M, (E)$1.0^{x}10^{-2}$ M, (F)$1.0^{x}10^{-1}$ M.CPE ingredients, (7% Ionophore ,35,50% Graphite powder ,46.50% Plasticizers (Di Butyl phthalate) ,7 % NaTBP, 3% Nio, and 1% graphene oxide**).**

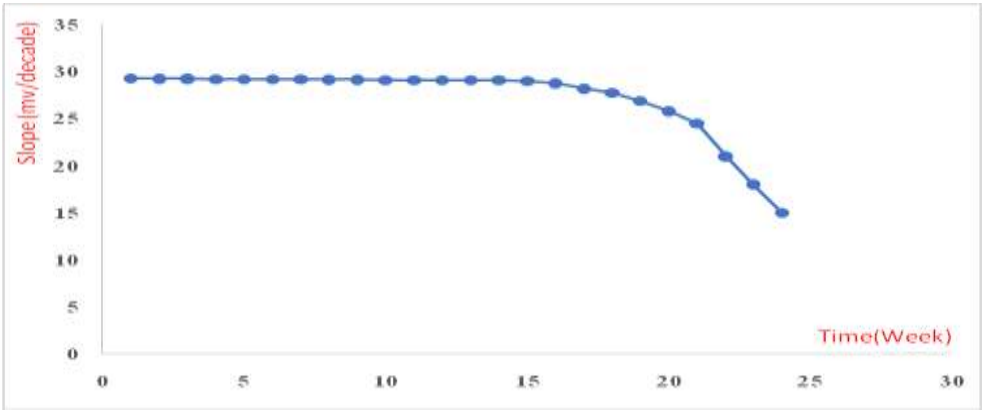


Figure (6): Life time of the Carbon Paste Selective Ni (II) electrode- ($_{CPE\text{-}Ni\ (II)Lp\text{-}Azo}$), CPE ingredients, (7% Ionophore ,35,50% Graphite powder ,46.50% Plasticizers (Di Butyl phthalate) ,7 % NaTBP, 3% Nio, and 1% graphene oxide

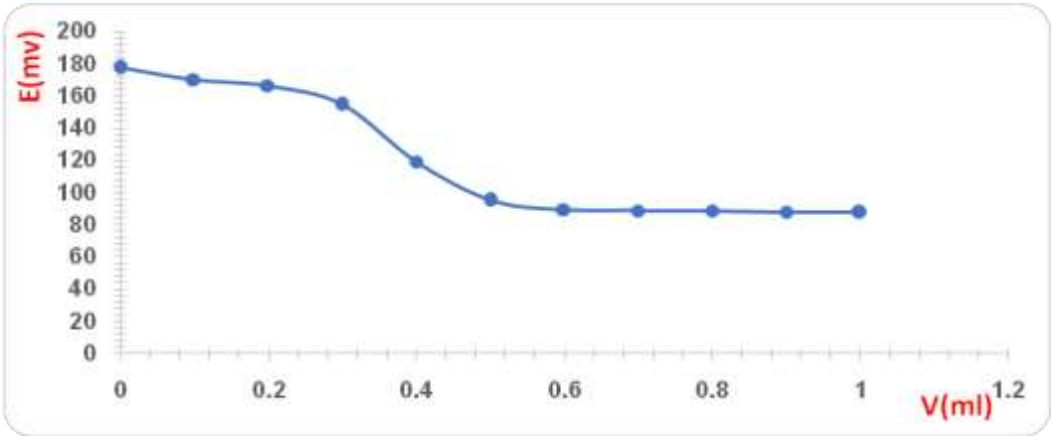


Figure (7): Potentiometric titration curve of 25 ml of Ni (II) ions (2.0^{x} 10^{-4}M) with standard solution of EDTA ($1.0^{x}10^{-2}$ M), using the proposed CPE-Ni (II)$_{Lp\text{-}Azo}$, CPE-Ni (II)ingredients, (7% Ionophore ,35,50% Graphite powder ,46.50% Plasticizers (Di Butyl phthalate) ,7 % NaTBP, 3% Nio, and 1% graphene oxide).

4. CONCLUSION:

In This work, Carbon Paste Selective Ni (II) electrode- **($_{CPE\text{-}Ni\ (II)Lp\text{-}Azo}$)** as a recognition element of an all-solid-state potentiometric Sensor. The proposed Sensor Showed a Nernstian response toward Ni^{2+}Species in an extensive concentration and pH range. The main advantages of the constructed electrode are simplicity of its preparation, short conducting time, fast response time a wide dynamic range, low detection limit, low cost long lifetime with a Nernstian response. The proposed electrode reveals an excellent selectivity towards Ni (II) ions in present of some of alkali, alkaline earth and some heavy metal ion, in aqueous medium. The proposed **[CPE-Ni (II)Lp-Azo**] was used successfully for potentiometric titration of this metal ion with EDTA.

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