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Determination of chloride (Cl^-) ion in water from Madurai region, Tamilnadu by Mohr's method

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Abstract- Surface water, bore well water and mineral water contain chloride ions at low concentration levels; chloride ion determination is carried out by titration with silver nitrate ($AgNO_3$) as titrant. The chloride ion (Cl^-) is a type of anion which is commonly found in the environment and it has paired with number of cations and involved in important physiological functions and industrial uses. However, a higher concentration of Cl^- in water will do harm to the ecological environment, human health and industrial production. There is a great significance to monitor the level of Cl^- ion content in water. Recent development of society and industry, large amounts of domestic sewage and industrial sewage are discharged into the environment, hence the water polluted by Cl^-. The detection of Cl^- in water has gradually become a research focus. In the present investigation we collected few water samples from Madurai region, Tamilnadu and determined the amount of chloride (Cl^-) ion by Mohr's method.

Keywords- method, titration method, Argentometric method

I.INTRODUCTION

Drinking water purity, availability and their nature not the only problem, in addition quality of water is also important factor. Adult human body has 60% water, the major portion of the body is covered by water, and no one replace the water equivalent. The nature of clean drinking water is colourless, odourless, and tasteless, not contain any chemicals and etc. The function of water in our body is infinite number and also regulating body temperature. Water has polar properties hence few dissociated in water to form ions.

These alkaline metal ions not involved in redox reaction under normal circumstances, but presence of excess salt chloride can lead to decrease the quality of water [1]. Hence such chloride (Cl^-) ion containing water was not suitable for irrigation and domestic life. The chloride is used for number of useful purposes. For example the addition of chlorine to water leads to render it "germ free". The chlorine is used as efficient disinfectant to kill disease causing [3]. Trichloromethanes are the main disinfectant by-products generated from chlorination with two different types of organic compounds, bromoform and dibromochloromethane. Continuous exposure of both bromoform and dibromochloromethane can cause liver, kidney, cancer, as well as heart diseases [4]. The world health organization (WHO) has pointed out that risk to health by these by-products is extremely small in comparison with the risk associated with inadequate disinfection [5]. The origin of chlorine in drinking water carried out from natural sources, sewage and industrial effluents and saline intrusion.

The chloride ion intrusion in human body was mainly from addition of salt to food and excess intake of salt containing drinking water [6]. The corrosion of metals in the water distribution system depends on the alkalinity of the water and excess chloride concentration. Higher concentrations of chlorides are leads to increase rate of corrosion of metallic pipes and structures. Chloride can be removed from drinking water by techniques like distillation, reverse osmosis or electro-dialysis. The number of technology is available for chloride treatment, but it is not possible on the basis of economical sense. Chlorides are harmless at low levels. The high concentration of sodium chloride in water can damage plants if used for gardening or irrigation. Because of sodium chloride high corrosivity over time there will be a damage plumbing, appliances, and water heaters, causing toxic metals to leach into your water.

There is no federally enforceable standard for chlorides in drinking water, but the EPA (Environmental Protection Agency) recommends levels not higher than 250 mg/L to avoid salty tastes and undesirable odors. The level of chloride (Cl$^-$) ion concentration greater than 250 mg/l sodium chloride can complicate existing heart problems and contribute to high blood pressure.

The chlorides can easily be removed from water with either a reverse osmosis system or a distiller. Reverse osmosis works by passing water through a semi-permeable membrane that separates pure water into one stream and salt water into another stream. In regular osmosis water flows from a lower concentration of salts to higher concentrations; in reverse osmosis the application of pressure greater than the osmotic pressure reverses the water flow from higher concentrations to much lower concentrations, producing pure water [7].

Background:

Chloride in the form of chloride (Cl$^-$) ion is one of the major inorganic anions in water and wastewater. Near the sea coast area chloride may be present in high concentration because of leakage of salt water into the sewage system, and also it may be increased by industrial process. In potable water, the salty taste produced by chloride concentration is variable and depends on the chemical composition of water. Waters containing 250 mg/L Cl$^-$ may have a detectable salty taste if sodium cation is present. The typical salty taste may be absent in waters containing as much as 1000 mg/L may be due to the presence of cations like calcium and magnesium. The measured chloride ions can be used to know salinity of different water sources [8].

II. MATERIALS AND METHODS

1. Chemicals and glassware: silver nitrate, sodium chloride, potassium chromate were purchased from Hi-media laboratories Pvt Ltd, Mumbai, India. Standard model burette, Pipette, beaker and conical flask were taken from our college laboratories for this experiment.

2. Water samples: Water samples for chloride ion determination were collected from Madurai region, Tamilnadu.

3. Solutions and reagents:

1. Standard sodium chloride solution (0.01 M): Dissolve 0.0584 g of AR grade NaCl (Equivalent weight = 58.44 g/mol) in distilled water in a 100 ml standard measuring flask, make up the solution to the mark with distilled water and homogenize. Silver nitrate solution (0.01 M): Dissolve 0.3398 g of AR grade silver nitrate (Equivalent weight = 169.87 g/mol) in 200 ml standard measuring flask with distilled water and homogenize. Potassium chromate solution (5% solution): Dissolve 5 g of AR grade potassium chromate in 100 ml of distilled water in a reagent bottle. Calcium carbonate: Use AR grade calcium carbonate powder.

2.

4. Principle: The water contains chloride (Cl$^-$) ions in the form of NaCl, KCl, $CaCl_2$ and $MgCl_2$. The maximum advisable limit of chloride ion in water is 250 ppm and above which the water is not desirable for drinking purpose. The amount of chloride ion in water can be determined by Argentometric or Mohr's method.

4. Procedure

1.Standardization of silver nitrate: All the glass apparatus first washed with tap water and then rinse thoroughly with distilled water. Rinse the burette with silver nitrate solution and fill it with the same solution up to zero mark. Pipette out 20 ml of standard sodium chloride solution into a clean 250 ml conical flask and add 1 ml of potassium chromate indicator. Titration of the above solution with silver nitrate solution taken in the burette until the first appearance of permanent red brown colour is formed. The faint reddish brown colour should persist permanently. Repeat the titrations for concordant values. From the titration values calculate the strength of silver nitrate solution.

2.Determination of chloride ion (Cl^-) concentration in the water samples: Pipette out the 20 ml of the collected water sample into a clean 250 ml conical flask. Add 1 ml of potassium chromate indicator and titrate the water sample solution against silver nitrate solution taken in the burette as per the procedure described in part A. From the titration values calculate the concentration of chloride ion in the water.

5. Calculations:

(i) Calculate the concentration of silver nitrate solution

V1 x N1 = V2 x N2
V1 = Volume of sodium chloride solution
N1 = Normality of sodium chloride solution
V2 = Volume of silver nitrate solution
N2 = Normality of silver nitrate solution
N2 = V1 x N1/ V2

(ii) Calculate the concentration of chloride (Cl^-) ion in the water sample.

N2 x V2′ = V3 x N3
N2 = Normality of silver nitrate solution
V2′ = Volume of silver nitrate solution
V3 = Volume of chloride (Cl^-) ion containing water sample
N3 = Normality of chloride (Cl^-) ion containing water sample
N3 = V2′ x N2/V3

The amount of chloride (Cl^-) present in 1 litre of water sample = N3 x equivalent weight of Cl^- ion

= N3 x 35.45 g/l

(iii) Formula

Weight/litre = Normality x equivalent weight
Weight/20 x 1000 = N3 x 35.45 g/l
Weight = N3 x 35.45/50 g/l
Weight = N3 x 35.45x 1000/50 mg/l or ppm

6. Theory involved detecting the end points in precipitation reaction [9]

Chloride (Cl^-) ion in water is determined by Mohr's method or precipitation titration. In this method chloride (Cl^-) ion is neutral or slightly alkaline solution is determined by titrating it with a standard solution of silver nitrate using potassium chromate as internal indicator. The pH of the sample must be in the range of 7-8, the reason being: (i) Ag (I) gets precipitated as AgOH at pH greater than 8. (ii) CrO_4^{2-} transforms into $Cr_2O_7^{2-}$ at pH less than 7.

At lower pH, $HCrO_4^{1-}$ being a weak acid, CrO_4^{2-} ion concentration decreases requiring higher concentration of Ag (I) for the solubility product of Ag_2CrO_4 to be exceeded thus leading to higher results. However, in many cases, the required pH can easily be maintained by adding a pinch of pure $CaCO_3$. In practice neutral solution of chloride taken in the conical flaskis directly titrated with standard silver nitrate solution taken in the burette using potassium chromate solution as internal indicator. As the titration is continued, silver ions combine with both chloride and chromate ions forming the respective precipitates of silver chloride and silver chromate.

$$Ag^+ + Cl^- \rightleftharpoons AgCl\downarrow \qquad (K_{sp} = 1.8 \times 10^{-10})$$

White precipitate

$$2\,Ag^+ + CrO_4^{2-} \leftrightharpoons Ag_2CrO_4\downarrow \qquad (K_{sp} = 1.1 \times 10^{-12})$$

Reddish brown precipitate

But, the red colour formed due to Ag_2CrO_4 by the addition of each drop of Ag^+ disappears because of the large concentration of Cl^- ions in the solution.

$$Ag_2CrO_4 + 2\,Cl^- \leftrightharpoons 2\,AgCl\downarrow + CrO_4^{2-}$$

Reddish brown precipitate White precipitate

During the course of the titration as the concentration of Cl^- ions decreases, the red colour disappears more slowly and when all the chlorine has been precipitated, a faint reddish or pinkish tinge persists in the solution even after brisk shaking.

7. Solubility product and precipitations involved in Mohr's process [10]

The process involved here is fractional precipitation between two sparingly soluble salts, ($K_{sp} = 1.2 \times 10^{-10}$ for AgCl; $K_{sp} = 1.7 \times 10^{-12}$ for Ag_2CrO_4). In actual practice the titration of NaCl with $AgNO_3$ was carried out in the presence of dilute K_2CrO_4 solution. AgCl is less soluble salt and the initial chloride concentration is high; hence AgCl will be precipitated. At the first point where red silver chromate is just precipitated both salts will be in equilibrium with the solution. Hence;

Solubility product of AgCl = 1.2×10^{-10} M
$[Ag^+]\,[Cl^-] = K_{sp}\,[AgCl] = 1.2 \times 10^{-10}$ M
Solubility product of Ag_2CrO_4 = 1.7×10^{-12} M
At the end point the concentration of both $[Ag^+]$ and $[Cl^-]$ reach an equilibrium.
$K_{sp}\,[AgCl] = [Ag^+]\,[Cl^-]$
At the end point
$[Ag^+] = [Cl^-]$

So, $K_{sp} = [Ag^+]^2$ or $[Cl^-]^2$
$[Cl^-]^2 = K_{sp}$
$[Cl^-] = \surd K_{sp} = \surd 1.2 \times 10^{-10} = 1.1 \times 10^{-5}$ M
At the end point both AgCl and Ag_2CrO_4 salts are in equilibrium. So,
$K_{sp} [AgCl]/[Cl^-] = \surd K_{sp}[Ag_2CrO_4]/[CrO_4^{2-}]$
$[Cl^-]/\surd[CrO_4^{2-}] = K_{sp}[AgCl]/ \surd K_{sp}[Ag_2CrO_4]$
$= 1.2 \times 10^{-10}/\surd 1.7 \times 10^{-12} = 9.2 \times 10^{-5}$ M
To form a precipitate when the concentration of Ag^+ is 1.1×10^{-5} M the concentration of chromate ion is
$[Cl^-]/\surd[CrO_4^{2-}] = 9.2 \times 10^{-5}$ M
$\surd[CrO_4^{2-}] = [Cl^-]/9.2 \times 10^{-5}$ M
$[CrO_4^{2-}] = \{[Cl^-]/9.2 \times 10^{-5}\}^2$
$= \{1.1 \times 10^{-5}/9.2 \times 10^{-5}\}^2$
$= 1.4 \times 10^{-2}$ M (or) 0.014 M
Thus Ag_2CrO_4 precipitate when chromate concentration is greater than 0.014 M and silver ion concentration less than 1.1×10^{-5} M.

III. RESULTS AND DISCUSSIONS

The most familiar Mohr's method in which alkaline or alkaline earth chlorides react with silver nitrate in presence of indicator potassium chromate solution as indicator a simple and accurate method for chloride ion determination. The significance of chloride (Cl^-) ion present in the water is known to everyone. But the presence of higher concentration (> 250 ppm) leads to unwanted toxic problem, corrosion in the industry and etc. In the present investigation we collected water sample from the Madurai region and analysed for chloride ion concentration. The amount of chloride (Cl^-) ion determined in the collected water sample was reported in table 1.

Table 1 Amount of chloride ion present in the water sample collected in Madurai region Tamilnadu, determined by Mohr's method

S. No.	Water sample collected area	Amount of Chloride (Cl^-) ion, ppm
1.	S. Vellaichamy Nadar College, Tap water	0.3858
2.	S. Vellaichamy Nadar College, RO water	0.2204
3.	Commercial 'Rivo' RO water	0.2204
4.	Rajapalayam, kanmai water Chokkanathanputhur,	1.2766
5.	Koviloor, Well water (kinaru)	2.8940
6.	Koviloor, Farm land water	2.5815
7.	Panniyan, bore water (G)	10.7798
8.	Panniyan, bore water (O)	8.5104
9.	Corporation water, Rajapalayam, Chokkanathputhur	0.2837

The collected RO (Reverse Osmosis) water for drinking purpose has lesser amount of chloride ion (0.220 ppm). But in the bore water and well water slightly higher amount (10.77 ppm) than the RO water. In Madurai region the water having lesser amount of chloride ion concentration, hence it can be utilized for the drinking purpose without any harm.

IV. CONCLUSIONS

The above study clearly reveals that the ground water and ponds water in Madurai region is not polluted by chloride. The lower level of chloride concentration in groundwater is clear indication of ground water not polluted with sewage water. With the continuous development of technology, chloride ion detection methods are also developing, but the simple titration method is used here to detect the chloride ion concentration. The analysis technology for Cl^- ion in water sample plays an important role, because it is the basis for the detection of Cl^- in food, feed, activated carbon, paper products, chemical products, fertilizer, air, soil, cement, and so on. The monitor of Cl^- ion is of concern to quality of soil, water, and air, the survival of animals and plants, human health, the safety and security of construction and building, the prediction of the corrosion of industrial equipment, industrial process control, the accuracy of chemical oxygen demand analysis, and so on., therefore, the detection of Cl^- ion in water is very important.

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