Research Article

Determination of Nitrite in Meat Samples Using Iodometric Titration Method

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ABSTRACT

In this work determination of nitrite was performed using iodometric technique. A qualitative analysis was carried out for nitrite determination. Conditions such as [KI],[NO_2^-] and pH were optimize. Disapperance of bluish green colour was taken as the end point. Out of different concentrations, for [KI]=5mM and 2.5mM,linearity in the graph (Equiv.[$Na_2S_2O_3$] Vs [NO_2^-]) plotted was observed. For the real sample analysis, four real samples such as Hen flesh, Hybrid variety hen flesh, flesh of goat and sheep flesh, with [KI] = 5 mM and [$Na_2S_2O_3$] = 5 mM were taken. Increase in concentration of [KI] shows a corresponding increase in the reaction. The real sample values are noted down, initially the points are found to be linear and then saturates.

Keywords: Potassium Iodide, Meat samples, $Na_2S_2O_3$.

1. INTRODUCTION

Nitrate is returned to the environment in animal faces, as well as through microbial degradation of plants and animals after they die. Micro organisms can convert nitrate or a ammonium ion to nitrate and this reaction occurs in the environment as well as within the digestive tract of human and other animals. After the bacteria convert nitrate to nitrite in the environment, the nitrogen cycle is completed when they then convert the nitrite to nitrogen. Normally this natural cycling process does not allow excessive amounts of nitrates or nitrites to accumulate in the environment. It is well known the nitrite is a reactive chemical and must be used with caution. It is lethal to humans in a dose of approximately 1g. Nitrite that can interfere with the oxygen transport system in the body and may result in the condition known as methaemoglobinaemia, in which the ability of hemoglobin to exchange oxygen is seriously reduced .Infants under 3 months are thought to be more susceptible than adults. Nitrite also acts as a nitro sating agent and under appropriate conditions produces nitroso compounds, some of which are specific and potent carcinogens. Furthermore, nitrite can be converted to nitric oxide, an active nitrosating agent, which can react with secondary amines and tertiary amines to form carcinogenic nitrosamines.

Due to these toxic efforts, it is important to develop new analysis methods for determination of nitrite in food products. Many analytical methods for the determination of nitrite have been developed.

In the current work, determination of nitrite was reported using iodometry technique. Conditions such as pH, [KI] and [NO_2^-] were optimized. Formation of linearity from the graphs plotted with Equiv.[$Na_2S_2O_3$] Vs [NO_2^-] validates this methodology. Further, the method was extended to determine the nitrite concentration present in different meat samples. Thus this method is highly appreciable for nitrite determination from various sources. The advantage of this method is simple, less expensive and easy to operate.

2.EXPERIMENTS

2.1 Apparatus: All apparatus used were of analytical grade obtained from borosil.

1. Conical flask (250 ml)
2. Burette (50ml)
3. Pipette (10ml)
4. Standard flask (50 ml, 100 ml and 250 ml).

2.2 Reagents

All reagents used were of analytical reagent grade obtained from E-Merck. Deionised distilled water was used to make up solutions for all physical measurements. Standard solutions of nitrite in pH=2 (KCl-HCl) Solutions. Meat and chicken products are purchased from local markets.

2.3 METHODS

2.3.1 Determination of Nitrite

To determine the nitrite is exactly 10 ml of standard sodium nitrite (2mM to 10mM) solution is pipetted out into a clean conical flask after rinsing the pipette with the sodium nitrite solution. A drop of starch indicator is added to it. Exactly 10ml of KI solution pipette out into a clean conical flask after rinsing the pipette KI solution. This work is preparing the KI solution various concentrations are taken for this titration Such as 15mM, 10mM, 5mM and 2.5mM. The solution becomes bluish green in colour. It is titrated against Equiv.[$Na_2S_2O_3$] solution 5mM taken in the burette.

III V

$$NO_2^- + 2I^- + ½\ O_2 \rightarrow I_2 \uparrow + NO_3^- \text{ (pH -2KCl-HCl)}$$

$$I_2 + 2Na_2S_2O_3 \rightarrow 2NaI + 2Na_2S_4O_6 \text{ (with starch indicator)}$$

The end point is the disappearance of Bluish green colour. The burette reading is noted. The titration is repeated for concordant value. The many concentration of NO_2- solution was titrated for concentration of Equiv. [$Na_2S_2O_3$] in [KI] solutions. The values are collected, the linear plots of Equiv. [$Na_2S_2O_3$] Vs [$NaNO_2$] with slope almost very close to unity in all the systems were studied. This method is calculated for the slope, regression and intercept. In this case the concentration of NO_2^- is slowly increase and also end point also increase so linearity is formed. The graph is obtained show a linear increase initially for the concentration of 2mM to 100 mM and slowly decreased.

2.3.2 Real sample analysis procedure

2.3.2.1 Sample preparation

Four real sample viz., hen flesh (sample#1), hybrid variety hen (sample#2), flesh of goat (sample#3) and sheep flesh (sample#4) were weighed for 5g each. A 0.1M concentration of $NaNO_2$ was weighed and added to all the flesh samples equally and stored in the ice-box overnight. The samples were then taken out and washed with copious amount of pH -2KCl-HCl buffer solution to collect 50ml of the real sample + $NaNO_2$ mixture. 5mM stock solutions of KI and Equiv.[$Na_2S_2O_3$] were prepared and 10ml of the KI solution was added to the above said real sample mixture. The KI added real sample mixture was taken for titration against Equvi. [$Na_2S_2O_3$].

2.3.2.2 Procedure for standard addition method

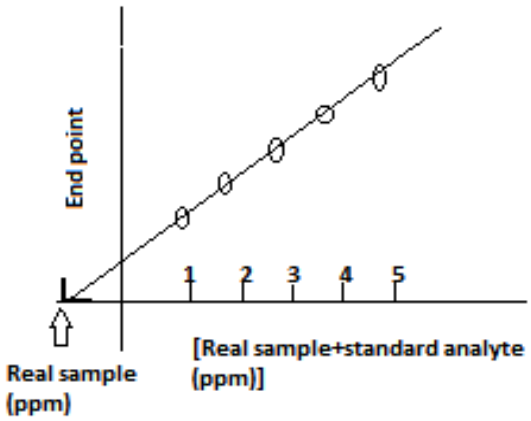
In this standard addition approach, known amount of the analyte standard solution were added with the real sample. From the real sample calibration plot, one can determine the amount of analyte present in the real sample.

The results obtained are tabulated as follows

Real sample + standard solution (50mL net)

	VOL. OF REAL SAMPLE (mL)	Analyte stock mL(Z)
1	y	Y+nZ
2	Y	$Y+n^1Z$
3	Y	$Y+n^2Z$
4	Y	$Y+n^3Z$
5	Y	$Y+n^4Z$

Where n = amount of the real samples added.



Plot for standard addition method

First collect the real samples from the testing place and prepare different real sample + standard analyte solutions(of variable concentrations) of total volume 50 mL mentioned in the above table. Each sample is then subjected to titration and the end point values were noted down. As plotted in the graph, the end point and the real sample analyte data were further taken in to graph. The intercept line in the x-axis will give the quantitative detail of the concentration of the analyte present in the meat samples.

2.3.2.3 Titration method using standard addition method

Four real sample were introduce the titration method. Exactly 10 ml of real sample mixture is pipetted out in to a clean conical flask after rinsing the pipetted with real sample mixture solution and KI (5 mM) solution is added, and standard [$NaNO_2$] solution is added for 10 ml. A drop of starch indicator is added to it. The solution becomes bluish green colour. It is against Equvi.[$Na_2S_2O_3$] taken in the burette. The end point is disappearance of bluish green colour. The burette reading is noted the titration is repeated for concorent value various concentration (10 mM, 1 mM ,0.1 mM and 0.01 mM) of real sample are titrated for fixed concentration Equvi. [$Na_2S_2O_3$] and [KI].

3. RESULT AND DISCUSSION

3.1 Qualitative analysis

Initially the presence of NO_2^- from the solution were determined qualitatively by optimizing suitable PH buffer solution varing from pH -1 to pH -7 were prepared. A standard amount of [KI] = 10 mM was added to the pH solutions and to that an equimolar [NO_2^-] was added. The formulation of yellow colour was taken as identification to propose the presence of NO_2^- in the solution.

The equimolar mixture solution of nitrite and iodide in various pHs of range 1-7. Yellow colour appeared for the solution in the pH window of 1-3, while it was colourless in pH 4-7. Formation of the yellow colour was highly rapid. Key species involved in the coloration is nitrous acid, HNO_2, since NO_2^- has an acid-dissociation constent, pKa value of 3.3.It has been reported that in acidic solution the nitrous acid reacts selectively with iodide to form iodine, which can give raise to yellow coloured solution.

$$2NO_2^- + 2I^- + 4H <=> 2NO + I_2 + 2H_2O \qquad \text{(Fast chemical reaction)}$$

Important observation of this work is selectively. The above reaction was found to be highly selective per sec, the yellow colour was found only in the presence of NO_2- and not for other common biological and other organic and inorganic compounds containing functional groups such as amine (-NH_2), carboxylic acid (-COOH), nitrate(NO_3^-) and chloride (Cl-) etc., by testing with potassium nitrate , ascorbic, citric acid , catechol , cysteine , dopamine , glucose , sulphide , thiourea and urea .

3.2 Real sample analysis by standard addition method

Four real sample are introduce the titration method.

Fig.a shows the plot for Equiv. [$Na_2S_2O_3$] Vs R_1 + [NO_2^-] (R_1 = Hen flesh) and fixed (KI =5 mM) the end point values are noted down. Initially the points are found to be linear and then saturates.

Fig.b shows the plot for Equiv. [$Na_2S_2O_3$] Vs R_2 + [NO_2^-] (R_2 = Hybrrid variety hen) and fixed KI =5 mM. The end point values are noted down. Initially the points are founds to be linear and saturates.

Fig.c shows the plot for Equiv. $[Na_2S_2O_3]$ Vs R_3 + $[NO_2^-]$ (R_3 = Flesh of goat) and fixed KI =5 mM. The end point values are noted. Initially the points are down found to be linear and the saturates.

Fig . d shows the plots for Equiv. $[Na_2S_2O_3]$ Vs R_4 + $[NO_2^-]$ (R_4 = Sheep flesh) and fixed KI = 5 mM. The end points values are noted down initially the points are found to be linear and then saturates.

The reason for the saturation may be due to the real sample matrix effect. Further details quantitative analysis is needed to eliminate matrix effect and for the determination of $[NO_2^-]$ from the actual real sample.

Plot for real sample using standard addition method

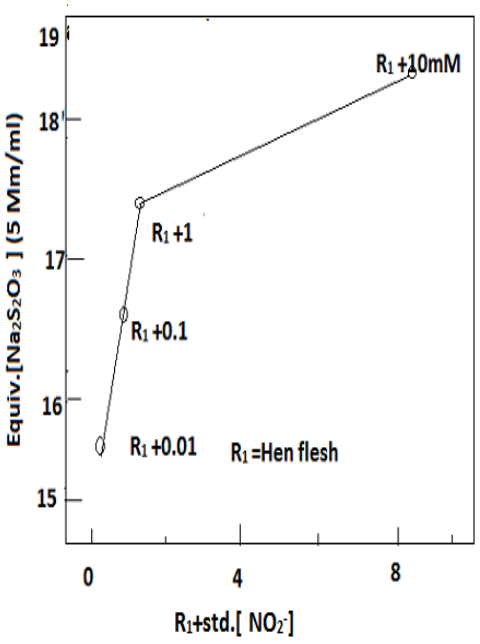


Fig.a

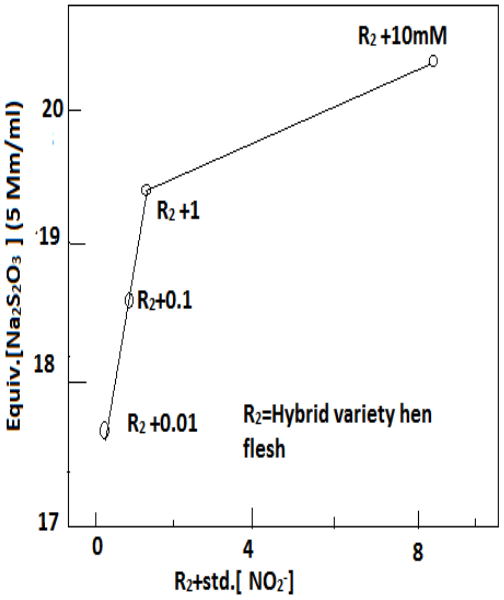


Fig.b

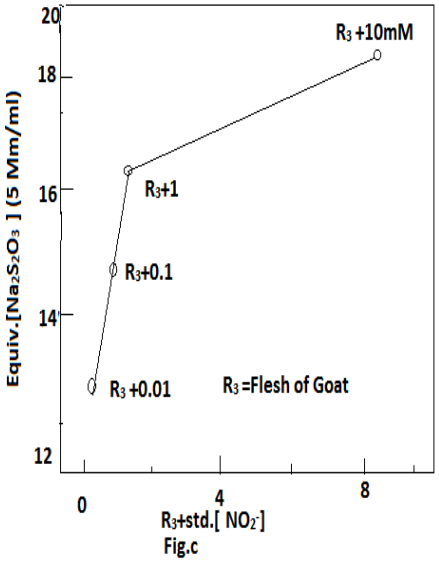
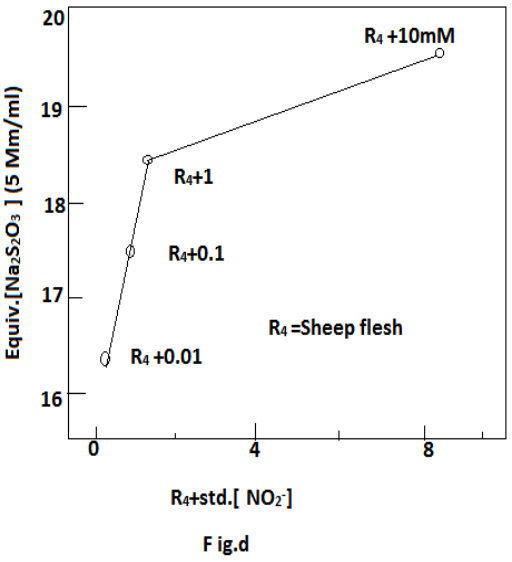


Fig.c



F ig.d

Table 1: Real sample analysis using standard addition method (Hen Flesh – sample #R_1) [$Na_2S_2O_3$] = 5 Mm

S. no	Real sample (sample# R_1)/mM	Concentration of KI in 10 ml(M)	Equvi. [$Na_2S_2O_3$] in (end point) ml
1	10	0.01	18.5
2	1	0.01	17.5
3	0.1	0.01	16.7
4	0.01	0.01	15.2

Table 2: Real sample analysis using standard addition method (Flesh of Hybrid varity Hen – sample #R_2) [$Na_2S_2O_3$] = 5mM

S. no	Real sample (sample# R_2)/mM	Concentration of KI in 10 ml(M)	Equvi. [$Na_2S_2O_3$] in (end point) ml
1	10	0.01	20.0
2	1	0.01	19.3
3	0.1	0.01	18.6
4	0.01	0.01	17.4

Table 3: Real sample analysis using standard addition method (Flesh of Goat – sample #R_3) [$Na_2S_2O_3$] = 5mM

S. no	Real sample (sample# R_3)/mM	Concentration of KI in 10 ml(M)	Equvi. [$Na_2S_2O_3$] in (end point) ml
1	10	0.01	18.0
2	1	0.01	16.2
3	0.1	0.01	14.5
4	0.01	0.01	13.0

Table 4: Real sample analysis using standard addition method (sheep flesh – sample #R_4) [$Na_2S_2O_3$]=5 mM

S. no	Real sample (sample# R_4)/mM	Concentration of KI in 10 ml(M)	Equvi. [$Na_2S_2O_3$] in (end point) ml
1	10	0.01	19.5
2	1	0.01	18.4
3	0.1	0.01	17.7
4	0.01	0.01	16.0

4. CONCLUSION

In this work we have proposed new titration methodology for the detection of nitrite. We have used equimolar mixture solution of nitrite and iodide in the pH range 1-7. Yellow colour solution is appeared for the pH window 1-3, while it was colourless in pH 4-7. Hence pH-2 was optimized for further identification. Real sample analysis was carried out using standard addition method. The real sample such as Hen flesh (sample#R_1), Hybrid variety hen (sample#R_2), flesh of Goat (sample#R_3) and Sheep flesh (sample#R_4) were used for the analysis. Initially linearity in plots was matrix effect. Further detailed quantitative analysis is needed to eliminate the matrix effect and for the determination of [NO_2^-] from the actual real sample found and then saturates. The reason for saturation may be due to the real sample matrix effect.

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