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ANALYTICAL EVALUATION OF STANDARD AOAC METHOD (NON-CATALYTIC) AND ACCELERATED WIJS METHOD(CATALYTIC)FOR THE DETERMINATION OF IODINE VALUE OF EDIBLE VEGETABLE OILS

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ABSTRACT: In the present work, the only variation is the use of mercuric acetate as a catalyst to reduce the analysis time to 3 minutes for the measurement of iodine value of edible oil and fats, wherein mercuric acetate is directly used in the powder form. Compared with the wijs method (non-catalytic) and catalytic method in which it take 30 minutes and 3 minutes to finish determination reaction of IV. An attempt has been made to reduce the time of the Wijs method by use of mercuric acetate as a catalyst/accelerator. The iodine value of different vegetable oils such as oleic acid (Oe), sesame oil (Se), refined mustard oil (Rmu,Dhaara),Pure kacchighaani mustard oil (Pkgmu, Fortune), Kacchiganni mustard (Kgmu1 ,Fortune) and mustard (mu2 ,Patanjali)oils were determined by regular Wijs method for 30 minutes whereas when we apply catalytic Wijs method with use of 2 mg, 5 mg and 10 mg of mercuric acetate to perform as catalyst then it is reducing the time of analysis to 3 minutes. When catalyst is used the different values obtained for standard deviations are 0.27 for 2mg**,** 0.28for 5mg and 0.16 for 10 mg whereas 0.27 for non-catalyst addition. The results obtained in the present work are more % difference in IV of refined mustard oil (Rmu,Dhaara) and Pure kacchighaani mustard oil (Pkgmu,Fortune).

KEYWORDS: IV (Iodine Value), mercuric acetate**,** vegetable oils

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1. INTRODUCTION

Mustard oil extracted from seasonal crops Brassica nigera, B.Junea and B. hitra has enormous edible and non-edible uses in India. The oil is consumed after extraction from mustard seeds, generally without any further processing, high price and dark colour of oil

make it vulnerable for adulteration. But there are some distinct physical and chemical parameters like refractive index, specific gravity, iodine value, colour, essential content ,unsaponifable composition, acid value, Free fatty acid content, peroxide value ,P-anisidine value of the oil helps to determine its conformity as safe and standard edible oil by which the purity check of mustard oil can be done. The official methods for determination of iodine value (IV) involve the reaction of double bonds in oils with halogenating reagent (Hanus or Wijs solution) over 30 min followed by iodometric titration of the liberated iodine obtained through reaction of excess Wijs reagent with potassium iodide with sodium thiosulphate solution using starch as an indicator. Wijs method is generally adopted for the measurement of iodine value [1,2].Various methods are available to determine the IV of fats and oils, such as that of Hanus and Hubl, Hofmann and Green, RosenmundandKuhnhenn and margosches method. The IV of vegetable oils can provide very useful information in other scientific fields. Although many methods have been developed, the Wijs method is the most widely used as a standard methodin food analysis laboratories. Major drawbacks of that method include the use of dangerous iodinetrichloride (Wijs reagent) and the time-consuming asduration of the reaction is as long as 30-60 minutes and procedures for reagent preparation and chemical analysis. Numerous efforts have been made to reduce the time by use of accelerators as mercuric acetate.

Generally Wijs method is used for measurement of iodine value and this method has a drawback that duration of the reaction is as long as 30-60 minutes. In this paper mercuric acetate is used as a catalyst/accelerator to achieve a reduction in the reaction time.

2. RELATED WORK

Mukherjee,S.(1955) investigated and developed a rapid method for the estimation of unsaturation of fats and oils by use of an aqueous solution of sodium hypochlorous acid reagent as a absorption reagent with a reaction time of 4 to 5 minutes was recommend, the estimations are more rapid or all drying or non-drying group oils give accurate results within the specified time [6].**Shin-ichiKikuno et al (1975)** investigated the methods of quick determination of iodine value especially for the oil in the hydrogenation process and have found after all the Wijs method could be appropriate by only shortening the reaction time to three minutes for the oils of iodine value less than about 100. It also studied the effect of catalyst, temperature, time and I/CL ratio during the determination of iodine value [7].**Hashemy et al (1977)** studied the IV of 121 samples of butter as well as some common

oils and fats by applying both the standard and rapid Wij's and Hanus methods. In the rapid method a 2.5% of mercuric acetate in acetic acid was used. The results obtained are close and comparable for 1 min Wij's and 3 min Hanus methods as compared with 30 min reaction time of standard procedures [4].

According to the united states patent (**1981**) when the magnesium acetate or sodium acetate is used in the form of a solution in glacial acetic acid, preferably having a concentration of 3-5 wt. %. In this method; the reaction time of a sample with the Wij's solution is as short as short as about 3 minutes. Then, the iodine value is measured in the same manner as in the Wijs method. Since The analysis time is thus remarkably shortened [9].**Lihua et al (1999)** investigated a fast method for determining the IV of oils and fats using mercuric acetate without changing the operational steps of the Hanus method and reduced time from 30 minutes to 3 minutes. The experimental result indicates that fast method gives a variation coefficient is 0.31 % [5].**Zhongguo-ging (2004)** investigated a new method for the determining the IV of oil and fat was only requires to add catalyst mercuric acetate in the process of determination without changing the operational procedure of Hanus method to reduce the reaction time of 30 minutes to 4 minutes. The experimental results indicate that the relative error is lower than 0.5 % and coefficient of variation is lower than 0.2% [10].**According to Yang Li,Ji Dong-bing et al (2014)** investigated the improved determination method was tested by adding Wijs reagent and 10 ml 3% magnesium acetate solution as catalyst reacting for 13 min., Acc. The result showed that there was no great difference between 2 methods with relative error less than 2%. It indicated that catalyst magnesium acetate had no adverse effect on accuracy of determination results [9].

Objective of thestudy is to develop a method by which time of the Wijs method can be reduced by use of mercuric acetate as a catalyst/accelerator. This research work aims at establishment of rapid, reliable and economical method for determination of IV of vegetable oils and examines the comparison between catalytic or accelerated method with original or non-catalytic AOAC Wijs method for IV analysis.

3. MATERIAL AND METHODS

3.1 **Procurement of Materials**

Vegetable oils such as oleic acid (Oe), sesame oil (Se), refined mustard oil (Rmu,Dhaara) , Pure kacchighaani mustard oil (Pkgmu,Fortune),Kacchiganni mustard (Kgmu1 ,Fortune)and mustard (mu2 ,Patanjali) oils have been purchased from the local market and

used in the present study for the determination of iv analysis.All these oils were in different forms of packaging while some in poly packs (HDPE), others were in tetra packs, plastic bottles, cans, pet and glass bottles of 1 liter and 5 liters. Since these eight different brands of edible oils were easily available for procurement (Table 1). Most of the brands have mentioned nutritional values, green vegetarian logo and best before 6 and 9 months, free from argemone oils on their packs. These different cooking oils are used in the present study for the determination of IV analysis.All the chemicals and reagents used in present experimental methodology are analytical grades.

3.2 Methods

3.2.1 Experimental Methodology

In the present work, use of mercuric acetate catalyst, and the absorption reaction time has been reduced to 3 min. It is the purpose of this paper to present a rapid method for estimation of unsaturation of oils. It provides accelerated catalytic method for the measurement of iodine value, wherein mercuric acetate is directly used in the powder form. The methodology includes addition of Wijs solution to a sample in an ordinary manner and then a powder form of the catalyst is added. The iodine value for a sample is determined in three set of experiments with 2 mg, 5 mg and 10 mg of mercuric acetate as a catalyst. The sample is allowed to react with the Wijs solution for reaction time about 3 minutes and then the iodine value is measured in the same manner as in the Wijs method.

3.2.2 Experimental procedure for determination of IV is according to Wijs method [2,3].

An appropriate amount of dry oil/fat as per expected IV (0.2-0.22mg) sample weighed into a tared Erlenmeyer flask with glass topper to which 25ml of carbon tetrachloride have been added and agitated for proper mixing. To this was added 25 ml Wijs reagent and mercuric acetate. The sample was evaluated in three set of experiments with 2 mg, 5mg, and 10 mg of mercuric acetate as catalyst. The flask was fitted with glass stopper wetted with KI solution, swirled for proper mixing and kept in a dark for about 3 minutes for reaction. The test was also performed in absence of mercuric acetate where it was kept in darks for 30 minutes. Simultaneously a blank test was also performed. At the end of reaction, to the flask was added 15 ml KI solution followed by 100 ml freshly boiled and cooled water with rinsing of the stopper. Liberated iodine was titrated with standardised sodium thiosulphate solution(0.1N) to reach pale yellow end point, nearly 2ml ml starch indicator

was added to flask and continue titrating the blue colour just disappears after through shaking(usually white end point). The iodine value was determined as follows:

Iodine value = 12.69* (B-S)* Normality of $Na_2S_2O_3$ /Weight of Sample taken

Table 1.1 Reports the iodine value of different vegetable oils determined by regular Wijs method and by the catalytic Wijs method with use of 2 mg, 5 mg and 10 mg mercuric acetate.

Table 1.1 IV Analysis of IV of vegetable oils by non-catalytic and catalytic Wijs method with reaction time of 30 and 3 min

Sr. No.	**Brand**	**Code no.**	**Name of oils**	**Expected IV**	**Use no catalyst**	**Use the catalyst**			**% Difference between catalytic and non-catalytic Method**		
					Reaction time						
					30 min.	**3 min**			**2mg**	**5mg**	**10mg**
						(2mg)	**(5mg)**	**(10mg)**			
				(a)	**(b)**	**(c)**	**(d)**	**(e)**			
1	-	Oe	Oleic acid	90.0	90.16	85.43	87.32	88.04	5.25	3.15	2.35
2	Tilsona	Se	Sesame oil	103-120	108.34	102.87	104.18	105.94	5.05	3.84	2.22
3	Dhaara	Rmu	Refined Mustard oil	96-112	105.34	97.98	99.76	102.12	6.99	5.30	3.17
4	Fortune	PKgmu	Pure KacchiGhaani Mustard oil	96-112	106.87	99.34	100.86	103.14	7.05	5.62	3.49
5	Fortune	Kgmu1	KacchiGhaani Mustard oil	96-112	107.27	102.18	103.57	105.22	4.75	3.45	1.35
6	Patanjali	Mu2	Mustard oil	96-112	104.66	100.12	102.51	103.76	4.34	2.05	0.86

Table-1.2 Accuracy of Iodine value in use of the catalyst and no catalyst

Sr.No	oil/fats	Use the Catalyst			Use no Catalyst
		IV (2mg)	IV (5mg)	IV (10mg)	IV
1	Oe	85.43	87.32	88.04	90.16
2	Se	102.87	104.18	105.94	108.34
3	Rmu	97.98	99.76	102.12	105.34
4	PKgmu	99.34	100.86	103.14	106.87
5	Kgmu1	102.18	103.57	105.22	107.27
6	Mu2	100.12	102.51	103.76	104.66
7	Total	587.92	598.2	608.22	622.64
8	Mean	97.99	99.7	101.37	103.77
9	SD	0.27	0.28	0.16	0.27
10	CV	0.28	0.28	0.16	0.28
11	SEM	0.22	0.22	0.13	0.22

Mean values ±SEM-standard error mean, SD-standard deviation, CV-coefficient of variation, IV-Iodine value

4. STATISTICAL ANALYSIS

The data obtained from the experimental measurements and accuracy of IV for different Groundnut seeds oils have been analysedand the Statistical parameter like standard deviation and coefficient of variation were calculated for IV.All the experiment was carried out in triplicateand **t**he results are presented as the mean SD,CV and SEM. Accuracy of descriptive Statistics of different groundnut oils from different parts of India as shown in figure1to3.

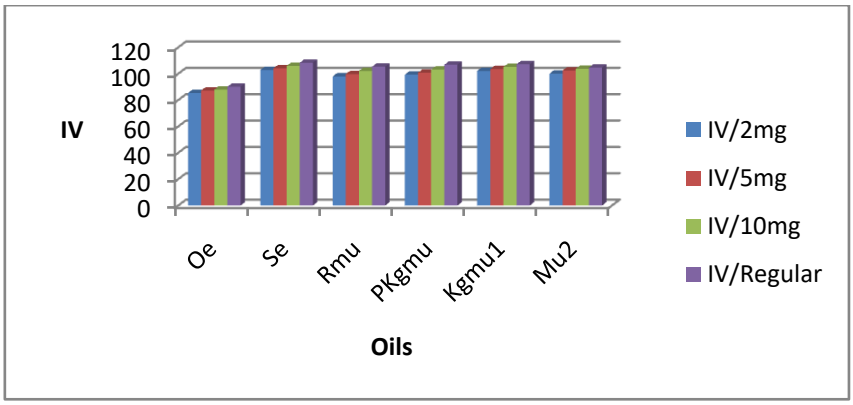

Figure1 Shows comparison of IV between reaction time of 30min(Regular) and 3min using 2mg,5mg and 10mg mercuric acetate catalyst

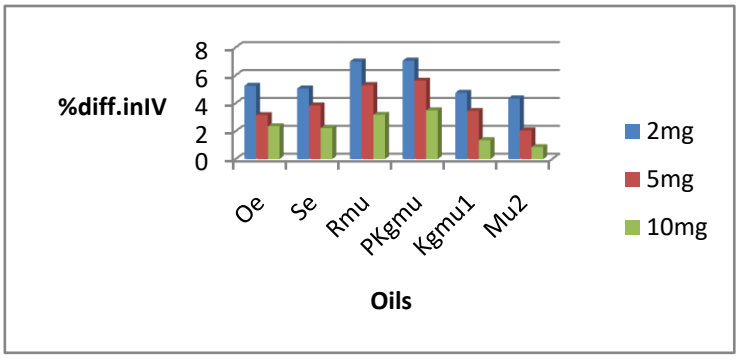

Figure 2 Shows comparison between % difference in catalytic and non-catalytic IV in 3min using 2,5 and 10mg of mercuric acetate

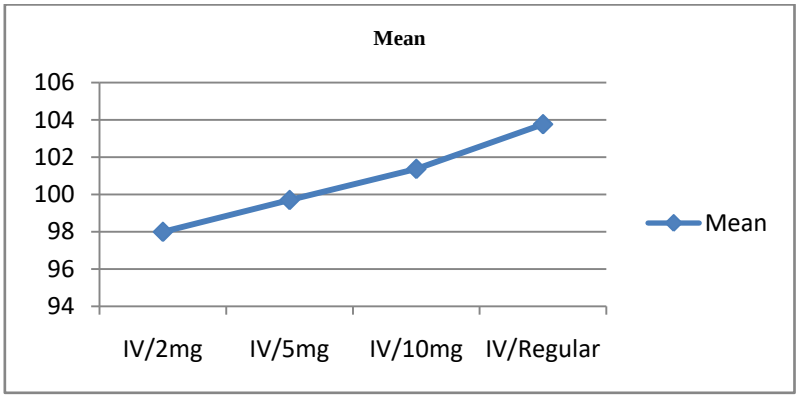

Figure 3 shows the accuracy of descriptive statistics of different brands of groundnut oils in India.

5. RESULTS AND DISCUSSION

It is apparent from the **Table 1.1** that the iodine value for oil/fat obtained by the Wijs method and by the experimental method (modified Wijs method) is not significantly different except Rmu&Pkgmu. All the experimental values are average of five readings with good reproducibility of results. Also results obtained by use of mercuric acetate lies within the expected range, as per Food safety and standards act 2006 and Food product and Standards regulation 2011 [column (a) of Table] [**3]** , of iodine value for respective oil/fat. The presence of catalyst has facilitated the increased reaction rate with reduction in time of analysis. It is observed that with increase in the quantity of catalyst reduces the difference in iodine value obtained by regular Wijs method and modified Wijs method. Accordingly use of 10mg of mercuric acetate gives least variation in the values obtained for all the studied oil samples. Comparatively more difference is noted in case iodine value by Wijs method and modified Wijs method for pure kacchighani mustard oil (Pkgmu) and refined mustard oil(Rmu), wherein the allowed time of 3 minutes is not sufficient for reaction between iodine monochloride and pure kacchighani mustard oil and refined mustard oil. This has however reduced with the increase of catalyst quantity. Higher time of reaction may favour the reduction in difference in values of IV by regular Wijs method and modified Wijs method. The obtained value of IV for all studied samples by modified Wijs method represents the success of mercuric acetate to perform as catalyst in reducing the time of analysis to 3 minutes. Moreover, as all the reported values are average of three readings, has demonstrated the reproducibility of the analysis data. Table 1.2 shows the variance of the measured values of the method of setting it to 3 minutes. The coefficient of variation in case of 2mg is 0.28while in case of 5mg (catalyst addition) 0.28and in 10 mg, 0.16, even for non-catalyst addition, 0.28.

6. CONCLUSION

Present research examines the comparison between catalytic or accelerated method with original or non-catalytic Wijs method for IV analysis. The accuracy, reproducibility and validity aspect of IV analysis has been conducted using mercuric acetate catalyst. It is found that there is no significant difference between the IV obtained by this catalytic method and standard AOAC method. This present work introduces significant reduction in

the analysis time, measurement accuracy and reproducibility of data for the determination of non-catalytic and catalytic IV analysis. Thus as a result catalytic Wijs method can be adopted as online quality control technique for rapid analysis during hydrogenation of oils and fats. The use of 10 mg of mercuric acetate gives least variation in the values obtained for all the studied oil samples.

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