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Measurement of Iodine Value (Wij's Method) of some Edible Vegetable Oils available in Market by using Accelerator

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Abstract: ***In the present work, an improved method is used for the determination of iodine value (IV) and to develop a method by which time of the Wijs method can be reduced time of analysis by use of mercuric acetate as a catalyst/accelerator. Wherein mercuric acetate is directly used in the powder form. 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 refined imported soyabean (Sib, fortune), refined soyabean (Sb1,Nature fresh,),refined soyabean (Sb2,Gemini) and refined soyabean (Sb3,fortune), refined soyabean (Rsb, Sundrop), refined groundnut (Rgn, Dalda), refined palmolein (Rpm1,Ruchi gold) and refined soyabean (Rsb1, Mahakosh) 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. The analytical results obtained with this procedure were compared with those from Wijs' method. When catalyst is used the different values obtained for coefficient of variations are 0.03 to 0.47 for 2mg, 0.02 to 0.54for 5mg and 0.02 to 0.54 for 10mg whereas 0.02 to 0.84 for non-catalyst addition. When we apply mercuric acetate as a catalyst/accelerator, the advantages obtained with the present work are significant reduction in the analysis time, measurement accuracy and reproducibility of data.***
Keywords: IV (Iodine Value), Wij's method, mercuric acetate, edible vegetable oils

I. INTRODUCTION

The quality of fats and oils is dictated by some distinct physical and chemical parameters like refractive index, specific gravity, iodine value, colour, 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 vegetable oil can be done. These can be dependent on the source of oil; geographic, climatic, and agronomic variables of growth. Thus one must assess quantitatively the influence of these variables on characteristics of oils and fats; in present case on characteristics of soyabean oil, Moreover vegetable oils from different geographical locations differs in oil content. In this case, 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 of 3 minutes.

For regular quality control(refining of hydrogenation plant) purposes as it requires around 30-60 minutes for the reaction of oils with the Wijs solutioni.e lengthy or time consuming for fast and non-destructive IV analysis of oils. The official methods for determination of iodine value (IV) involve the reaction of double bonds in oils with halogenating reagent (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]**and involves following reactions:

$$ICl + KI \longrightarrow KCl + I_2$$

$$I_2 + 2\ Na_2S_2O_3 \longrightarrow 2\ NaI + Na_2S_4O_6$$

A. Related Work

Hoffman and Green used mercuric acetate as a catalyst in the Wijs method to obtain complete iodine absorption in three minutes **[4]**. Benhen and Klee modified the Rosenmund-Kuhnhenn Method so that only one minute reaction time was required **[2]**.According to Jnmiat et al,various methods and modifications that have been proposed on time to time for the determination of the Iodine values of fats and oils, Hubl's and Wijs' are the only two methods which have found more or less general application**[3].**

S.mukherjee, investigated and developed a rapid method for the estimation of unsaturation of fats and oils by use of an aqueous solution of sodiumhypochlorous acid reagentas a absorption reagent with a reaction time of 4 to5 minutes was recommend,the

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estimations are more rapid or all drying or non drying group oilsgive accurate results within the specified time[6].Shin-ichiKikuno et al 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 alstudied 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 state 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%[10].

Several methods spectrophotometric methods have been developed for rapid determination of IV. The standards which used for constructing a calibration graph were fatty acids with the known IV obtained Wijs solution method by (AOAC, 2000)[1].

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%[11].Thidarat et al., (2012)proposed a spectrophotometric method has been developed for the determination of iodine value of vegetable oils and based on the reaction of Hanus solution with oils and subsequently treated with potassium iodide solution producing triiodide ions.

The absorbance at 350 nm of triiodide ions was used for analytical purposes. The calibration graph was constructed by plotting the absorbance at 350 nm versus the molar concentration of Hanus solution. Under the optimal conditions a linear calibration graph ranged from 0.02 to 0.10 mol/L of Hanus solution with R^2 of 0.999.The proposed method was feasible to determine a wide range of iodine value of vegetable oil samples.

The standard deviation of iodine value ranged from 0.7 to 2.1 (n=3).The analytical procedures were simple, required short analysis time, and used small amount of solvent and reagent[12].In addition, the methods required the standardization of oils or fatty acids used for construction of calibration graph by using the time consuming official methods.

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 3 min.,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[13].

Aim of the study is to develop a method by which time of the Wijs method can be reduced by use of mercuric acetate as a catalyst/accelerator. Present research also examines the comparison between catalytic or accelerated wijs method with original or regular Wijs method for IV analysis.

II. MATERIAL AND METHODS

A. Procurement of Materials

Vegetable oils such as refined imported soyabean (Sib,fortune), refined soyabean (Sb1,Nature fresh,),refined soyabean (Sb2,Gemini) and refined soyabean (Sb3,fortune), refined soyabean (Rsb, Sundrop), refined groundnut (Rgn,Dalda),refined palmolein (Rpm1,Ruchi gold) and refined soyabean (Rsb1, Mahakosh) oils have been purchased from the local market and 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 litre and 5 litres. 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.

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Table 1 Different oils purchased from local market of Jalgaon city

Sr.no	Name of oils	Brand	Place of manufacturer company	Product type/expiry*
1	Refined imported soyabean oil	Fortune	Adani wilmer limited (unit II)VIII, Dhrup, Tal- Mundra, Dist. Kutch Gujarat	Made from imported oils, free from argemone oils, contains permitted antioxidants and OMG-3,essential PUMA
2	Refined soyabean oil	Nature Fresh	Agro tech foods limited, Premier soya oil limited, Industrial area, Jhotwara-302012	free from argemone oils
3	Refined soyabean oil	Gemini	Cargill India private limited, VIII-Bhimsar, Kutch, Gujarat-148024	free from argemone oils, contains permitted antioxidants and OMG-3,essential PUMA
4	Refined soyabean oil	Fortune	Adani wilmer limited (unit II)VIII, Dhrup, Tal-Mundra, Dist. Kutch Gujarat	free from argemone oils, contains permitted antioxidants and OMG-3, essential PUMA
5	Refined soyanean oil	Sundrop	Agrotech foods limited, Premier soya oil limited, Industrial area, Jhotwara-302012	free from argemone oils, contains permitted antioxidants (TBHQ)
6	Refined groundnut oil	Dalda	Bunge India private limited JMD Oils private limited, Survey no.320, Bhimsar, Gandhi dam, Kutch-370110, Gujarat	free from argemone oils
7	Refined Palmolein oil	Ruchi gold	Ruchi Infrastructure limited,706, Tulsiani chamber, Nariman point, Mumbai	Made from imported oil , free from argemone oils
8	Refined soyabean oil	Mahakosh	Jain Agro oils, A-45,Matiala village, Uttam Nagar, New Delhi-110059	free from argemone oils

*All the oils have best before within 6 and 9 months from the packaging.

B. Methods

1) *Experimental Methodology:* In the present work, an attempt has been made to reduce the time of the Wijs method by use of mercuric acetate as a catalyst/accelerator. It provides a rapid 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.

2) *Experimental procedure for determination of IV using Wijs method[2,14]:* The only variation is the use of mercuric acetate as a catalyst to reduce the analysis time. To a 500ml Erlenmeyer conical flask with glass topper was weighed accurately an appropriate quantity of the dry oil/fat as per expected value (0.2-0.22mg), 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) using starch as indicator until the blue colour formed disappears after through shaking. The iodine value was determined as follows:

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

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Table 1.2 IV Analysis of IV of vegetable oils by Wijs method using 2 mg, 5 mg and 10 mg mercuric acetate with reaction time of 30 and 3 min

Sr. No.	Code of oils /fats	Expected IV	Use the catalyst			Use no catalyst	% Difference between catalytic and non-catalytic Method		
			Reaction time						
			3 min			30 min.			
			(2mg)	(5mg)	(10mg)				
		(a)	(b)	(c)	(d)	(e)	(e-b)*100/e	(e-c)*100/e	(e-d)*100/e
1	Sib	120-135	125.02	126.18	127.0	127.97	2.31	1.54	0.76
2	Sb1	120-135	124.83	125.27	127.6	128.32	2.72	2.38	0.56
3	Sb2	120-135	125.02	126.15	126.86	127.04	1.59	0.7	0.001
4	Sb3	120-135	124.65	125.88	127.12	128.62	3.9	2.13	1.17
5	Rsb	120-135	125.02	126.09	127.0	128.18	2.47	1.64	0.93
6	Rgn	85-99	85.83	88.71	90.55	91.27	5.96	2.8	0.79
7	Rpm1	54-62	54.02	55.17	56.26	58.15	7.1	5.14	3.25
8	Rsb1	120-135	124.33	125.76	126.80	128.98	3.61	2.50	1.55

Table-1.3 Accuracy of Iodine value in use of the catalyst and no catalyst

Sr. No	oil/fats	Use the Catalyst									Use no Catalyst		
		2mg			5mg			10mg					
		IV*	σ	CV (%)	IV*	σ	CV (%)	IV*	σ	CV (%)	IV	σ	CV (%)
1	Sib	125.02	0.14	0.11	126.18	0.09	0.08	127.0	0.29	0.23	127.97	0.66	0.51
2	Sb1	124.83	0.41	0.47	125.27	0.31	0.35	127.6	0.48	0.54	128.32	0.49	0.54
3	Sb2	125.02	0.21	0.39	126.15	0.3	0.54	126.86	0.23	0.41	127.04	0.49	0.84
4	Sb3	124.65	0.04	0.03	125.88	0.02	0.02	127.12	0.02	0.02	128.62	0.03	0.02
5	Rsb	125.02	0.14	0.11	126.09	0.09	0.08	127.0	0.29	0.23	128.18	0.66	0.51
6	Rgn	85.83	0.41	0.47	88.71	0.31	0.35	90.55	0.48	0.54	91.27	0.49	0.54
7	Rpm	54.02	0.21	0.39	55.17	0.3	0.54	56.26	0.23	0.41	58.15	0.49	0.84
8	Rsb1	124.33	0.13	0.11	125.76	0.14	0.11	126.8	0.16	0.12	128.98	0.16	0.12

*Average values of three measurements, σ-standard deviation, CV-coefficient of variation

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III. STATISTICAL ANALYSIS

The data obtained from the experimental measurements and accuracy of IV for different Groundnut seeds oils have been analysed and 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. Accuracy of descriptive Statistics of different groundnut oils from different parts of India as shown in figure1to2.

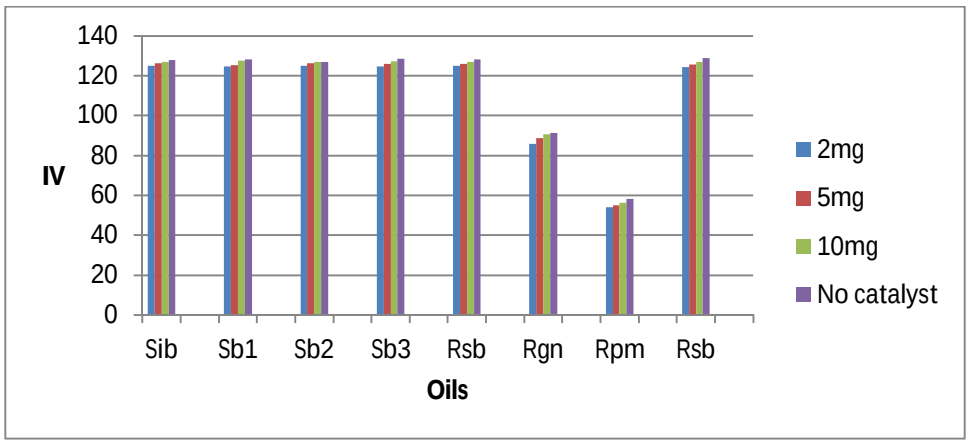

Figure 1.Shows comparison of IV between reaction time of 3min and 30min using 2,5 and 10mg mercuric acetate catalyst

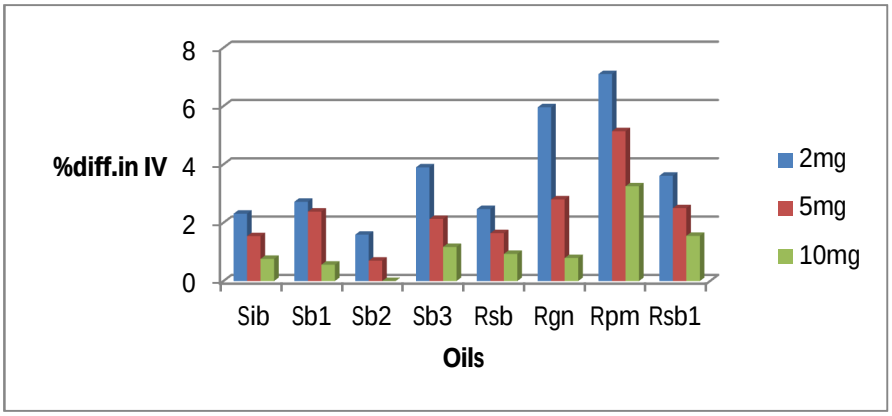

Figure2. Shows comparison between % difference in catalytic and non catalytic IV in 3min using 2, 5and 10mg of mercuric acetate

IV. RESULTS AND DISCUSSIONS

The 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][4], of iodine value for respective oil/fat.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. 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. Comparatively more difference is noted in case iodine value by Wijs method and modified Wijs method for refined Palmoleinoil (Rpm). 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 0.03 to 0.47while in case of 5mg (catalyst addition) 0.02 to 0.54and in 10 mg, 0.02 to 0.54, even for non- catalyst addition, 0.02 to 0.84.

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V. CONCLUSION

This present work introduces significant reduction in the analysis time, measurement accuracy and reproducibility of data for the determination of regular Wijs method and catalytic Wijs method for 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. 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.

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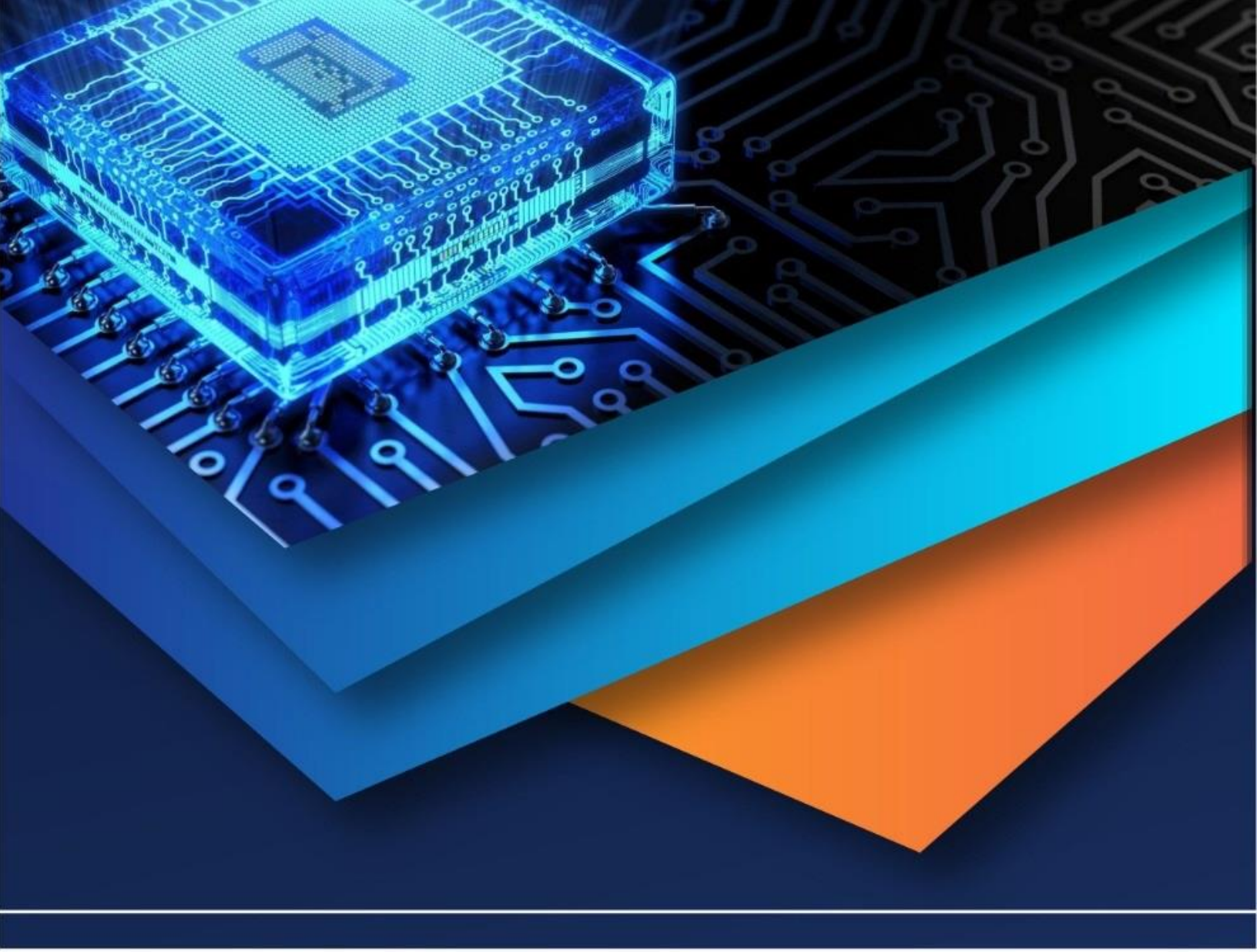

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