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Assessing the Genuinty of different Edible Cooking Oils available in Local Markets of India using Bellier Turbidity Temperature Test (BTTT)

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Abstract: ***Oils containing long saturated fatty acids give a precipitate at a particular temperature which is specific for the oil when their alcoholic soap solution is treated with dilute acetic acid solution and 70% ethyl alcohol. In this study an attempt has been made to investigate the applicability of BTTT to different edible cooking oils obtained from different parts of India and thereby examine the influence of geographical variations on BTTT. In the present work, the different brands of edible cooking oils used for analysis, such as refined cottonseed oil(Fortune plus,rct)(19.8), Pure cottonseed oil(Tirupati, pct)(20.9), refined groundnut oil(RRO primio,rgn)(39.8), filtered groundnut oil(Snehdrop, fgn)(40.2), filtered groundnut oil(Dammani,fgn1)(40.5), kacchi ghaani mustard oil(Panghat, mukg)(26.8),Premium til oil(RRO Tildil, pt)(19.9),Sesame oil (Shakti, Ss) (20.9),Nigerseed oil(Fortune, ngs)27.5),Kardai ghani oil(Jijau, kg)(15.6).*** The ***result have demonstrated the reproducibility through the analyzed data. Hence It is observed that cottonseed oil fulfils BTTT values as per Regulation (Food Products and Standards and food additives) 2011 of Food Safety Standards and Act 2006. The standard mean error is in between 0.06-0.12 in case of BTT. The BTTT method is cheaper, easier, requires little laboratory infrastructure and recognized as a convenient qualitative tool for identification of different varieties of oils.***

Keywords: Different brands of edible cooking oils, genuinty, BTTT.

I. INTRODUCTION AND OBJECTIVE

Edible oils had made an important contribution to the diet of people in many countries serving as a good source of protein, lipid, and fatty acids for human nutrition including the repair of worn out tissues and new cells formation as well as a useful source of energy [1]. Vegetable oils and fats have wide application in foods where they are used in frying, salad dressing, shortening of pasty, margarine, cooking, and ice cream manufacture. In the world, vegetable oils and fat are found to be about 80–85% of edible oils and fat consumed by the public [2]. Edible oils are very important food for the world. The human body uses oils and fats in the diet for three purposes, such as being an energy source, being a structural component, and making powerful biological regulators. Oils and fats also play an important role in metabolic reactions in the human body [3]. Edible oils are resulting from plants and animals. Oils from plants are termed as vegetable oil. The principal sources of vegetable oils are nearly plants, which include sunflower, soybeans, cotton seed, sesame, rapeseed, corn, melon and sesame seed. Other sources are oil bearing perennial plants such as shear, olive, cashew coconut and palm. Sesame seed is rich in oil content with about 53% quality edible oil, 42% cake and 5% moisture seed [4]. Fats or oils consist of a wide group of compounds that are soluble in organic solvents and insoluble in water. They have lower densities than water and at normal room temperature range inconsistently from liquids to solids depending on their structure and composition. The words oils, fats, and lipids are all used to refer to fat; oils are usually used to refer to fats that are liquids at room temperature, while fats are usually used to refer to that are solid at normal temperature. Lipids are used to refer to both liquids and solids fats [5].

In quality control of edible oils, several parameters such as iodine value (degree of unsaturation), saponification value (average molecular weight), moisture content, and peroxide value as well as the free fatty acid content are of interest as they determine the quality and hence the economic value of the product. Currently, the majority of these parameters are determined by using classical wet chemical methods [6].Several factors affect the edible oil quality such as agronomic techniques, seasonal conditions, sanitary state of drupes, ripening stage, harvesting and carriage systems, method and duration of storage, and processing technology. The major

Factors affecting edible oil quality are temperature, moisture, sunlight, soil fertility, and nutrients. It is possible to determine by different analytical techniques how to assess the quality of edible oil and to avoid possible adulterations [7,8].

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The quality of fats and oils is dictated by several physical such as texture, density, pacific gravity, colour, refractive index etc and chemical parameters such as acid value, iodine value, saponification value, unsaponifable matter BTT etc are 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 different edible oils, Bellier Turbidity Temperature Test (BTTT) (acetic acid method), based on insolubility of Arachidic acid is used as a qualitative method for identification of all the pure cooking oils used in this research work. Sometimes it is observed that groundnut and mustard oils fulfils all specifications of refined oil but fails to pass BTTT. The imposition of BTT values to raise the issue pertaining to the discrepancy in BTT for the presence of other vegetable oils (admixture of oils) in groundnut and mustard oils. Moreover all the varieties of oilseeds from different geographical locations differ in oil content. The Bellier figure or the temperature at which turbidity appears in a specified and neutralized oil sample under specified conditions was first proposed by Bellier and modified by several workers including Franz and Adler. According to Ever in 1912, the addition of sufficient acetic acid used instated of 1% hydrochloric acid succeeding modifications in the BTT. This had been adopted by several workers and gives satisfactory results for sufficient to judge the purity of peanut oil and admixture of oils. In most cases the Bellier figure increases with the % of peanut oil in the mixture. The increase is not proportional and there is a steep rise for the % of peanut oil below 25 % [9].

The objective of the present studies was to investigate the applicability of BTTT to different brands of different cooking edible oils obtained from different parts of India and thereby examine the influence of geographical variations on BTTT as tool for identification of different cooking oils.

II. LITERATURE REVIEW

The pea nut, often called as “The King of Oilseeds”, is botanically known as *Arachis hypogaea* and belongs to family Leguminosae, which is also called Fabacae. The pea nuts differ in the quantity as well the quality of oil. These differences in the pea nut oil may be due to several factors *i.e.* genotype, the level of maturity of the seed, season and geographical area of production **[10]**.About 80% of the total fatty acid content of peanut oil constitutes unsaturated fatty acids mainly oleic acid and linoleic acid **[11]**.Thus the chemistry and quality of pea nut oil mostly depend on the oleic to linoleic ratio. The studies observed that the oil containing high UFAs/SFAs ratios are thermodynamically more stable and may be heated to high temperatures **[12]**. The oil containing higher content of MUFAs fatty acids (oleic acid) are more stable to oxidative damage during refining and storage **[13]**.On the other hand, other scientist suggested that the linoleic acid, a PUFA, having two double bonds is more susceptible to oxidative rancidity than oleic acid as well as the saturated fatty acids. But Linoleic acid, being an essential fatty acid, also plays a beneficial role in human health in lowering the total blood cholesterol and LDL levels. The long term stability of peanut oil may also be associated with the antioxidant substances (tocopherols and polyphenols) present in peanut oil as the minor components **[14]**.

Mustard Oil may provide a protective effect in connection with patients having acute myocardial infarction, possibly due to the presence of α-Linolenic acid. It has been found that the omega-3 PUFA present in rapeseed/mustard oil reduces the risk of chemically induced cancer **[15]**.However; higher levels of erucic acid are unsuitable for human consumption. Oils having low erucic acid are recommended for human consumption because oils high in erucic acid may cause an accumulation of triacylglycerols in the hearts of animals. The major source of erucic acid is seed oils of the Crucifereae family, which includes rapeseed, mustard, cram be and wallflower. The erucic acid is known very crucial raw material for oleo chemical industry. Erucic acid and its derivatives possess varieties of superior properties in slipping, softening, antifoaming, emulsifying, and corrosion inhibiting. All these properties offer erucic acid and its derivatives wide applications in the production of pharmaceuticals, soaps, detergents, cosmetics, plastics, lubricants, rubbers, coatings **[16].**

Fatty acid profile of sesame oil showed that major component was linoleic acid containing 41.8–45.1% of the total fatty acids, followed by stearic 32.6–24%, palmitic 8.2–7. %, oleic 4.6–56% and these four comprised on 96% of the total fatty acids. About 83% of total means of linoleic and oleic acids were as unsaturated fatty acids of sesame. Sesame oil fit for human consumption because high amount of unsaturated fatty acids increases the quality of the oil. Saturated fatty acids of sesame oil were palmitic and stearic acids with a range of 9.1–10.4 and 3.2–5.9%. Phenolic compounds donate a hydrogen atom o serve up as significant antioxidants because of their donating ability in order to form stable radical intermediates. Hence, they phenolic compounds help in prevention the oxidation of different biological molecules **[17]**.

Gossypium hirsutumis is one of the most significant crops (fiber/food), native to tropical and subtropical regions in the world. After soybean, cotton is assumed as one of the most excellent source of plant (vegetable)protein and the fifth major seed oil crop after sunflower, canola, palm and soybean **[18]**.Cottonseed oil is obtained from the seeds of cotton plant and known as a by-product with about(12%) of the gross value of the total product. Cottonseed oil is usually utilized in cooking or frying and also used in some

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other industrial applications, whereas cakes after oil extraction are used in the preparation of poultry and animal feeds. Due to unique fatty acid profile, cottonseed oil is different among other vegetable oils as it holds a comparatively high level of unsaturation and considered as a healthy vegetable oil. Its fatty acid composition is distinctive of the oleic/linoleic group of vegetable oils, as these two fatty acids make up 73% of the total fatty acids (oleic acid and linoleic acid 17% and 56%, respectively) along with palmitic acid approximately (23%) **[19]**.

1) *Test for Presence of Cottonseed Oil (Halphen Test):* The development of red colour on heating the oil with a solution of sulphur in carbon disulphide indicates the presence of cottonseed oil. The test is also given by Hempseed oil, Kapokseed oil / oils and fats containing cyclopropenoid fatty acids (such as sterculic and malvalic acid). Hydrogenation and deodorization wholly or partially destroy the cromogens and react with diminished intensity. A positive reaction is not given by oil heated to 250°C or above. The fat of animals fed on cottonseed meal (butter, lard) or other cottonseed products may give faint positive reaction by this test. Take about 5 ml of the oil or melted fat in a test tube and add to it an equal volume of the sulphur solution (one percent (w/v) solution of sulphur in carbon disulphide and then add an equal volume of amyl alcohol). Mix thoroughly by shaking and heat gently on a water bath (70º to 80ºC) for a few minutes with occasional shaking until the carbon disulphide has boiled off and the sample stops foaming. Place the tube in an oil bath or a saturated brine-bath maintained at 110-115ºC and hold for 2.5 hours. A red colour at the end of this period indicates the presence of cottonseed oil. The test is sensitive to the extent of 0.5 % cottonseed oil in other oils. As per Food safety and standards (prohibition and restriction on sale) Regulations 2011, sale of certain admixtures prohibited. As per 2.1.1(5), a mixture of two or more edible oils as an edible oil ,a maximum tolerance of 10 red units in one cm cell on Lovibond tintometer scale is permitted when the oil is tested for halphen test without dilution. Halphen test is one of the qualitative identification tests for cottonseed oil [22].

As per Food safety and standards (prohibition and restriction on sale) Regulations 2011, sale of certain admixtures prohibited. As per 2.1.1(5), a mixture of two or more edible oils as an edible oil ,a maximum tolerance of 15 red units in one cm cell on Lovibond tintometer scale is permitted when the oil is tested for Baudouin test without dilution. Baudouin test is one of the qualitative identification tests for sesame oil. This test is sensitive to the extent of 0.2% of sesame oil in other oils[20].In this Modified Baudouin test, take 5 mL of the sesame oil or melted fat in a 25 mL measuring cylinder (or test tube) provided with a glass stopper, and add 5 mL of hydrochloric acid and 0.4 mL of furfural solution. Insert the glass stopper and shake vigorously for two minutes. Allow the mixture to separate. The development of a pink colour in the acid layer indicates the presence of sesame oil. Pink to red colour is obtained due to presence of a phenolic component sesamol [21].

Niger seed oil has linoleic acid (C18:2) as the principal fatty acid (65.7-68.5 %, weight percent
of total lipid). Oleic acid (C18:1) was the second major unsaturated fatty acid (5.4-7.5 %).Niger contains two major saturated fatty acids [palmitic (9.6-10 %) and stearic (7.6-8.1 %)].The above fatty acids represent 91-97% of the fatty acid present. Palmitoleic, linolenic, arachidic, eicosenoic, behenic, erucic and lignoceric acids constituted less than 1% each [23].The oil content of niger seed ranges of 40–44 %. Niger seed oil, like sunflower and nigerseed oils, contains high content of omega-6 PUFA i.e. linoleic acid (63-75%) [24]. Dietary fats and oils, rich in linoleic acid, have been reported to prevent cardiovascular disorders such as coronary heart disease, atherosclerosis, as well as high blood pressure. Also linoleic acid derivatives serve as structural components of the plasma membrane and as precursors of some metabolic regulatory compounds [25].

Safflower oil is thought to be one of the highest quality vegetable oils, containing oleic acid and linoleic acid. Standard safflower oil is a healthy oil with high amount of polyunsaturated fatty acid (PUFA), linoleic acid (>70%) and high ratio between polyunsaturated and saturated fatty acids among edible oils [26].Safflower is also a good source of monounsaturated fatty acid (MUFA), oleic acid (>70%) (Monounsaturated, MUFA) ,which is more stable and preferred for deep frying applications in the food industry[27].

The solubility of oils in various solvents is a constant, depending on the nature of the glycerides composing the oil. Fryer and Weston found that a mixture of equal volume of 92% ethyl alcohol and pure amyl alcohol used as a solvent for turbidity. In Valenta test, acetic acid was used as a solvent, the results are affected by the presence of moisture in the oil and free fatty acid which lower the turbidity temperature, increasing the solubility of the oils, which raises the turbidity temperature [9].

The modified BTT test has been used by Ever for judging the purity of oils and has been found simple, rapid and fairly accurate for routine analysis as compared to the results obtained by Valenta test. Moreover, it can be conveniently used in the analysis of soap and commercial fatty acids and also for determining the % of two mixed oils. Others workers have also successfully used the same test for determining adulteration of groundnut oil in some edible oils and also suggested its analytical importance. Besides the turbidity temperatures obtained with fatty acids by the method of fryer and Weston are different from those for the respective oils, depending on the difference in the solubility of the glycerides of the oil and its fatty acids in the same solvent [28].

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BTT test is useful to check purity of groundnut oil. BTT values for arachis (groundnut) oil depend on the relative insolubility of arachidic acid (C20:0) in 70% ethyl alcohol (1:2). The high BTT values of groundnut oil compared with the other vegetable oils is due to the insolubility of arachidic acid but due to the lignoceric acid (C24:0) present in the groundnut oil. They concluded that there is no direct relationship between the added lignoceric acid in groundnut oil which is responsible for the high BTT value. However, higher concentrations of lignoceric acid present in oil improve the perception of turbidity [29].

Table-1 Shows BTT standards/values for some edible vegetable oils under 2.2: Fats, oils and Fat emulsions as per FSSA 2006[20]

Sr.no	Item no	Vegetable oil	BTT limits	Arachidic acid(C20:0)
1	2.2.1.2	Cotton seed oil	19.0 -21.0^{0}C	0.4-1.3%
2	2.2.1.3	Groundnut oil	39.0-41.0^{0}C	1-2%
3	2.2.1.6	Rape seed oil Mustard oil (toria oil)	23.0-27.5 ^{0}C	0.5-2.4%
4	2.2.1.7	Rape seed oil or Mustard oil-Low erucic acid	Not more than 19.0^{0}C	
5	2.2.1.8	Virgin olive oil	17.0^{0}C Max	0-0.8%
		Refined olive oil		
6	2.2.1.10	Safflower seed oil (barrey ka tel)	Not more than 16.0^{0}C	0-0.34%
7	2.2.1.12	Til oil (Gingelly/sesame oil)	Not more than 22.0^{0}C	0.4-1.1%
8	2.2.1.13	Niger seed oil (sargiya ka tel)	25.0-29.0^{0}C	0-0.37%
9	2.2.1.17	Almond oil	Not more than 60.0^{0}C	0-0.43%

Source FSSA2006

III. MATERIAL AND EXPERIMENTAL METHODS

A. Materials

All the chemicals and reagents were analytical grade and used as received. Eight different cooking oils of different brands such as refined cottonseed oil(Fortune plus,rct), Pure cottonseed oil(Tirupati,pct), refined groundnut oil(RRO primio,rgn), filtered groundnut oil(Snehdrop,fgn), filtered groundnut oil(Dammani,fgn1), kacchi ghaani mustard oil(Panghat,mukg),Premium til oil(RRO Tildil,pt),Sesame oil(Shakti,Ss),Nigerseed oil(Fortune,ngs),Kardai ghani oil(Jijau,kg) oils were gathered from super market of different places of India. Since these four cottonseed oils were easily available for procurement. 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 100ml to1 liters and 5 liters. Since these eight different brands of edible oils were easily available for procurement. Most of the brands have mentioned nutritional values, green vegetarian logo and best before 6, 9 months and 12months, free from argemone on their packs. These different cooking oils are used in the investigations on BTTT in this research study.

B. Experimental Methods

1) *Determination of Bellier Turbidity Temperature Acetic Acid Method:* Pipette out one ml of the filtered sample of oil in a flat-bottom 100 ml round flask, add 5ml of 1.5 N alcoholic potash heating over a boiling water bath using an air condenser After complete saponification cooling, neutralized by adding carefully dilute acetic acid and then add an extra amount of 0.4 ml of accurately measured dilute acetic acid using phenolphthalein indicator. Add 50 ml of 70% alcohol and mixed well. Heat and allow the flask to cool in air with frequent shaking. Note the temperature by using calibrated thermometer at which the first distinct turbidity appears which is the turbidity temperature. This turbidity temperature is confirmed by a little further cooling which results in deposition of the precipitate. Dissolve the precipitate by heating the contents to 50°C over water bath, again cool as desiccated above and make a triplicate determination of the turbidity temperature [**21,22]**.

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Table 2: BTTT of different cooking oils with accuracy on BTT

Sr.No	Name of oil	Brand name	Code	BTTT	BTT STD value	SD	CV	SEM
1	refined cottonseed oil	Fortune plus	rct	19.8	19-21	0.17	0.87	0.1
2	Pure cottonseed oil	Tirupati	pct	20.9	19-21	0.1	0.48	0.06
3	refined groundnut oil	RRO primio	rgn	39.8	39-41	0.2	0.5	0.12
4	filtered groundnut oil	Snehdrop	fgn	40.2	39-41	0.17	0.43	0.09
5	filtered groundnut oil	Dammani	fgn1	40.5	39-41	0.1	0.25	0.06
6	kacchi ghaani mustard oil	Panghat	mukg	26.8	23-27.5	0.17	0.65	0.09
7	Premium til oil	RRO Tildil	pt	19.9	22max	0.1	0.5	0.06
8	Sesame oil	Shakti	Ss	20.9	22max	0.1	0.48	0.06
9	nigerseed oil	Fortune	ngs	27.5	25-29	0.1	0.36	0.06
10	Kardai ghani oil (safflower oil)	Jijau	kg	15.6	16max	0.1	0.64	0.06

* Each value is averages of three measurements, SD-standard deviation, CV-coefficient of variance, SEM-Standard mean error

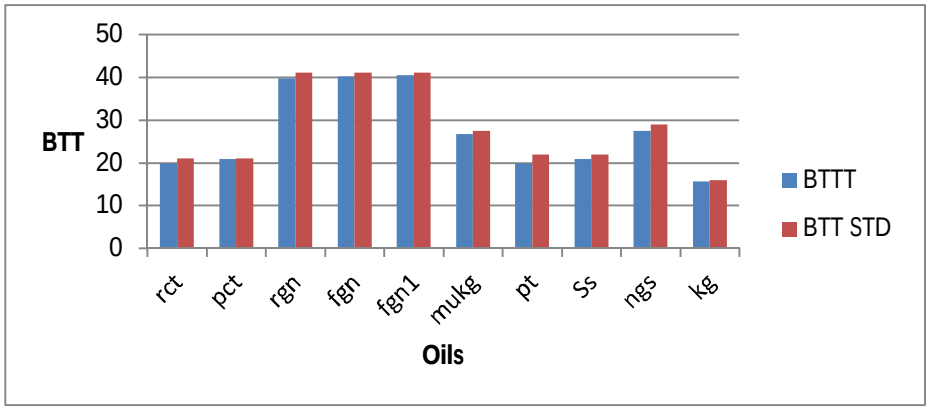

Fig.1 shows the comparison between experimental and standard BTTT values for different cooking oil

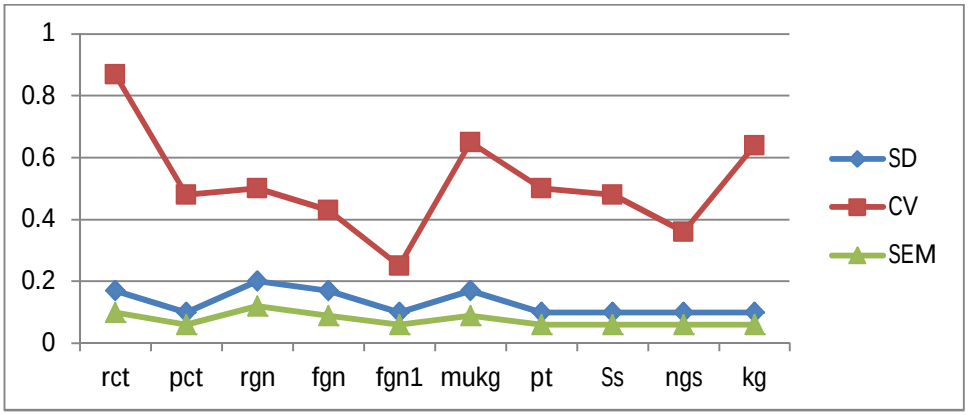

Fig.2 shows the Statistical values for different cooking oils

IV. STATISTICAL ANALYSIS

The data obtained from the experimental measurements and accuracy of BTTT for different brands of different edible cooking oils have been analyzed and the Statistical parameter like standard deviation, coefficient of variance and standard mean error were calculated for both the parameters. All the experiment was carried out in triplicate and the results are presented as the mean SD, CV and SEM as shown in Table2. Descriptive Statistics of different types of oils varieties from different parts of India as shown in figure1 and 2.

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V. RESULT AND DISCUSSION

BTT values prescribed for the certain vegetable oils comes under the mandatory food laws in some countries but due to development towards hybridization in oil seeds, reconsideration in laws is required. **Table-1** Shows BTT standards/values for some edible vegetable oils under 2.2: Fats, oils and Fat emulsions as per FSSA 2006**[20]**.The results obtained for the BTTT and statistical accuracy for the different cooking oils obtained from different places of India are shown in **Table2, Fig 1 and Fig 2**. The data obtained for rct(19.8), pct(20.9), rgn(39.8),fgn(40.2),fgn1(40.5),mukg(26.8),pt(19.9),Ss(20.9),ngs(27.5),kg(15.6) are exhibited BTT in the range of 15.6 to40.5°C.As all the reported BTTT values are average of three readings, the results have demonstrated the reproducibility of the analysis data. Thus the present investigations prove with due certainty the applicability of BTTT to all cooking oils. **Table 2** shows the accuracy, the standard deviation and coefficient is in the range of 0.1 -0.2 and 0.25-0.87.

VI. FUTURE PROSPECTS

Wherever required, BTTT analysis, Quantitative test should essential and can be easily supplemented with GC and HPLC analysis, which provide the quantitative data on presence of high molecular weight fatty acids in different cooking oils. Hence BTTT depends on the presence of arachidic acid and other higher acids in different cooking oil.

VII. CONCLUSION

The BTTT method is cheaper, easier, requires little laboratory infrastructure and recognized as a convenient qualitative tool for identification of different brands of oils available in local market. In this study BTTT is applied on different cooking oils and found that BTTT can be easily used as qualitative tool for identification of purity of all the different cooking oil from different places of India. The present investigations prove with due certainty about applicability of BTTT to all different cooking oils. This study also confirms prove reliability, reproducibility and diverse applicability of BTTT. BTT values prescribed for the certain vegetable oils comes under the mandatory food laws in some countries but due to development towards hybridization in oil seeds, reconsideration in laws is required.

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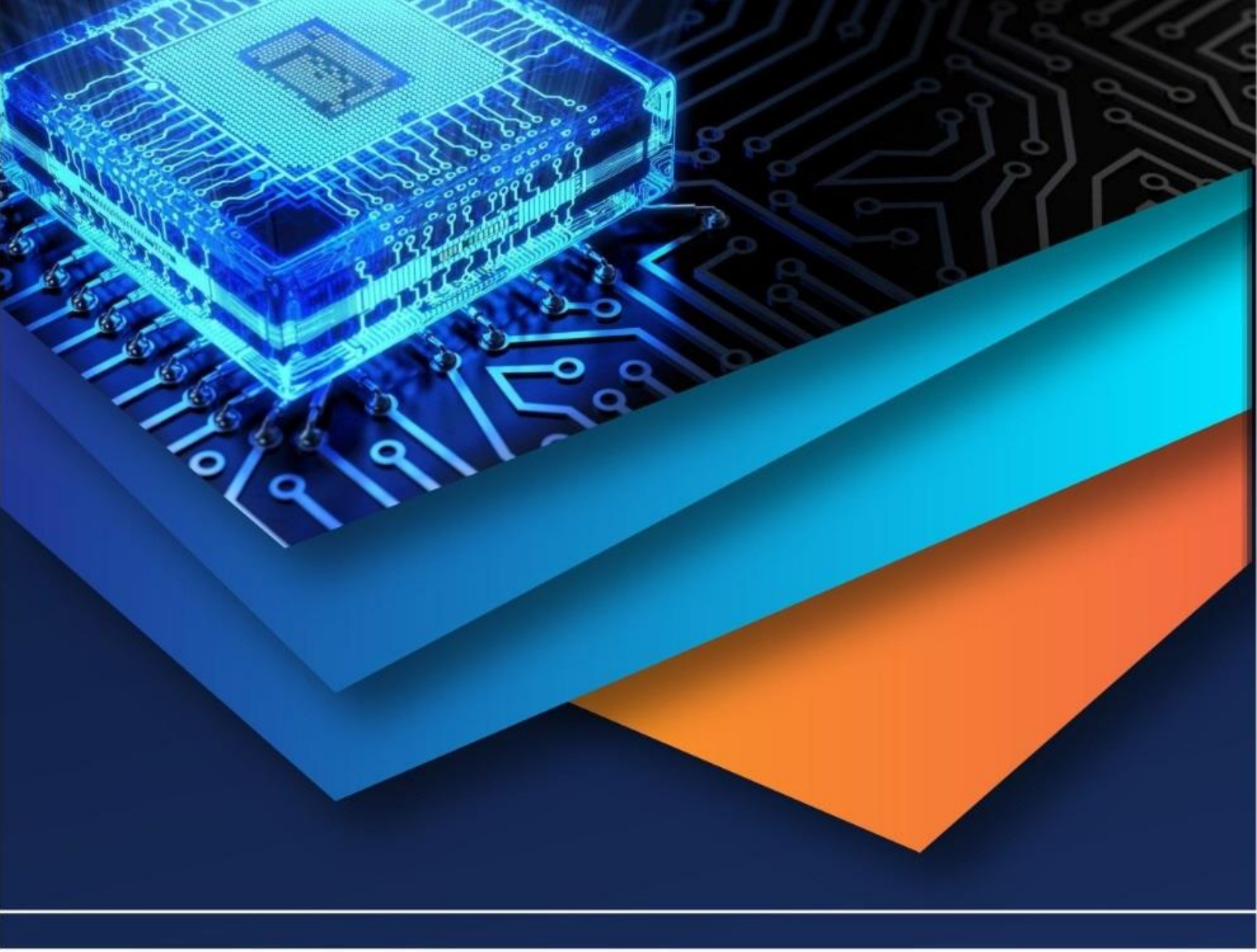

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