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Development and Method Validation for the Quantitative Estimation of Diclofenac Sodium Tablet by Spectrophotometric Method

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Abstract: ***Spectrophotometric method is used to developed and validated for quantifying and detecting the drug diclofenac sodium. The analytical process development and validation are essential for finding the easiest way to evaluate diclofenac sodium with maintain the safety, quality and effectiveness of pharmaceutical products. Ultra Violet (UV) spectroscopy is commonly used for substance analysis in the pharmaceutical industry due to its ability to deliver quick and accurate results. The technique measures the absorption of the ultra violet light, typically between 200-400 nm, making it effective for analyzing compounds based on their UV absorption characteristics. To meet regulatory requirements, the method must be validated for parameters such as specificity, precision, accuracy, reproducibility, limit of determination (LOD), and limit of quantification (LOQ) This ensures that the technique provides reliable and consistent measurements of active pharmaceutical ingredients and excipients, following established guidelines for optimal performance and quality control. This method follows the Beer-Lambert's Law. Accuracy of the drug was about the acceptance criteria as per the ICH guidelines.48***
Keywords: Diclofenac Sodium, UV Spectroscopy, Analytical process development, validation parameters, ICH guidelines.

I. INTRODUCTION

A. *Analytical chemistry*[1]

Analytical chemistry is a part of chemistry that deals with finding out what substance are in a sample and how much quantity of each substance is there. It aims on two main types of analysis.

1) *Qualitative analysis:* It tells us that which kind of chemicals or elements are in the sample.
2) *Quantitative analysis:*It states about how much quantity of each chemical or element is present.

There are mainly two methods those are used in this analytical chemistry-

- *Classical method:*These are older techniques that use simple tools and chemical reactions.
- *Instrumental method:*These use machines and advanced tools to study the sample.

B. *Spectroscopy*[2]

[It is a branch of science that refers how light or electromagnetic radiation interacts with matter. When this interaction happens, matter can absorb or give off energy in small, fixed amounts called quanta. Spectroscopy is very important method for getting on for the structure of atoms and molecules. It's also used to analyze many different kinds of samples. Optical spectroscopy focuses on the part of the electromagnetic spectrum that ranges from 100 angstroms (Å) to 400 micrometers (μm).

C. *UV Visible Spectrophotometer[3,4,5]*

A UV-Visible Spectrophotometer is a device used to measure how much ultraviolet (UV) and visible light a substance absorbs. It's working takes place by passing light through a sample and that measures how much light is absorbed at various wavelengths(200-800nm). Different substances absorb light differently, so this helps to identify what the substance is and how much of it is present. This tool is often used in labs to study chemicals, check purity, or measure concentrations of solutions.

The main rule used in spectrophotometric analysis is the Beer's –Lambert law.

- Beer's Law: This law states that when light passes by a substance, the additional molecules it has, the more the light gets absorbed. Hence, the amount of light is absorbed relies on the concentration of the substance.

- Lambert's Law: This law states that when light travels by a material, it gets weaker the farther distance it travels. The light absorption rises by the thickness of the material.
  Together, these two laws make up the Beer-Lambert law, which helps us understand how much light is absorbed based on the concentration and thickness of a substance.
- Beer's-Lambert law: It interpret how light is absorbed when it passes through a substance.

It says that the amount of light absorbed depends on: How concentrated the substance is (more concentration = more absorption),How thick the substance layer is (longer path = more absorption),And a constant value that depends on the substance itself.

In short: More concentration or thickness = more light absorbed.

The formula is–$\mathbf{A = \varepsilon \times c \times t}$[3]

Where,

A = Absorbance (how much amount of light is absorbed)

ε = Molar absorptivity (a constant)

c = Concentration of the solution

t = Path length (thickness of the solution, the light travels through)

D. Instrumentation [6,7]

1) *Light source:*Provides continuous radiation. Deuterium lamp (UV region: 200–400 nm)Tungsten-halogen lamp (Visible region: 400–800 nm).
2) *Monochromator:* Isolate a specific narrow band of wavelength of light. It concists of prisms or diffraction gratings to emit light.
3) *Sample Holder:* Holds the sample solution. Quartz cuvettes (for UV)Glass or plastic cuvettes (for visible light).
4) *Detector:*Sense and measures the intensity of light that is transmitted. Photomultiplier tube (PMT) (high sensitivity)Photodiode array (PDA) (rapid wavelength detection).
5) *Display & Data ProcessingUnit:* Records and analyses absorption data. This unit is connected to a computer for analysis of spectra.

These components work together to determine the absorbance or transmittance of a substance at a specific wavelength.

E. The Development of Method [8,9]

It means creating and improving a way or process to do something accurately and reliably. In analytical chemistry, method development refers to:

Figuring out the best steps to test or measure something (like the amount of a chemical in a solution),

Choosing the right tools and conditions (like temperature, time, or solvents),

Making sure the method gives clear, repeatable, and correct results.

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Making sure the method gives clear, repeatable, and correct results.

1) Understand the analyte
2) Define method goals
3) Research existing method
4) Select the best method
5) Set up instruments and run initial test
6) Fine-tune the method
7) Record key performance details
8) Test the method with real samples
9) Check sample recovery and accuracy

F. Method Validation [10]

Method validation is the process of confirming that a specific method, usually analytical or testing, is reliable, accurate, and suitable for its intended purpose. This is commonly done in scientific, pharmaceutical, medical, and technical fields. In a general sense, method validation ensures:

1) Accuracy – This method gives results similarly close to the true value.
2) Precision – When the process are repeated, the results are accurate.
3) Specificity – The method measures exactly what it is supposed to measure.
4) Sensitivity – It can detect even small amounts of the substance or signal
5) Linearity – Obtained results and concentration are directly proportional over a range.
6) Robustness – It remains reliable under small variations in conditions.
7) LOD & LOQ

G. *Drug Profile* [11,12]

1) *Structure:*

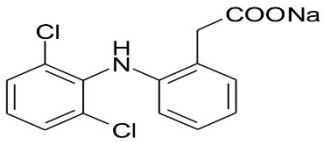


Fig. 1 Diclofenac Sodium

2) *Diclofenac Sodium:*It comes under the classification of nonsteroidal anti-inflammatory drugs (NSAIDs). It works by blocking cyclooxygenase (COX) enzymes and reducing the production of prostaglandins, which are accountable for pain and inflammation.
3) *Mechanism of Action:* Potent COX-2 inhibition: Diclofenac is considered one of the most potent COX-2 inhibitors among NSAIDs, making it a valuable tool for studying the role of COX-2 in inflammation and pain pathways. Wide range of applications:Researchers can study diclofenac's effects in various conditions like osteoarthritis, rheumatoid arthritis, musculoskeletal pain, migraine headaches, and even certain cancers due to its broad anti-inflammatory and analgesic activity.
4) *Chemical name:*2-[{2,6-dichlorophenyl}amino]benzene acetic acid
5) *Molecular Formula:*$C_{14}H_{10}Cl_2NNaO_2$.
6) *Molecular weight:*318.13.
7) *Route:* Oral.
8) *Pharmacokinetic Data:*Bioavailability-50–60%

- *Metabolism* - 99% in liver
- *Elimination half-life* - 3–6 hours
- *Excretion* - Urine and bile (90%)

II. AIM & OBJECTIVE

The aim of this project is to develop an analytical method and validate it for ensuring accuracy, precision, specificity, reliability, LOD & LOQ, of the analytical procedure. It confirms that this method produces compatible and reproducible results for the analysis of diclofenac sodium.

The parameters are defined below–

1) *Specificity*–Specificity is about how well a test can detect only the substance you're looking for (called the analyte), even when other things are in the sample.
2) For example, if you're testing for a certain drug in a tablet, specificity means the test can find that drug clearly—even if the tablet also contains other ingredients or impurities. It ensures no confusion between the target substance and anything else that might be present.
3) *Linearity*- Linearity refers to how well the test results match up with the amount of substance present. In simple terms, if you have twice as much of the substance, the test result should also be about twice as high. A good linear response helps in accurately measuring different amounts of the substance across a wide range.
4) *Range*-Range is the span between the smallest and the largest amounts of the substance for which the test works well. Within this range, the test should be: Accurate (gives correct results),Precise (gives consistent results), and Linear (results change proportionally with concentration).It tells us how much of the substance can be present and gives reliable results.

5) *Accuracy*- Accuracy describes us how close a measured result is to the true accepted value. Imagine weighing a 100-gram object—if your scale shows 100 grams, it's accurate. If it shows 105 grams, it's not. In analytical testing, accuracy ensures the measured amount is as close as possible to the real or accepted value.
6) *Precision*–Precision is about consistency. If you test the same sample several times and get very similar results each time, the method is precise. There are three levels:

- Repeatability:testing under the exact same conditions (same person, equipment, and time).
- Intermediate Precision:testing carried out under slightly changed conditions (different day, person, or equipment, but same lab).
- Reproducibility: testing in different laboratories altogether. A method can be precise but it cannot be accurate, so all are important.

7) *Intermediate Precision*- This is a part of precision. It shows how well the test performs when done under slightly different conditions within the same lab—like using a different analyst or doing the test on another day. It helps prove the method is reliable in day-to-day use, not just in ideal conditions.
8) *Robustness*-Robustness checks whether the test still works well even if there are small, intentional changes in how it's done. For example, slight changes in temperature, pH, or timing shouldn't affect the results too much if the method is robust. It shows that the test is dependable even when small variations happen, as they often do in real-world labs.
9) *LOD(Limit of Detection)*- LOD is the least amount of a substance or signal which can be distinguished reliably, from zero by a system of measurement. It tells us the lowest concentration at which the substance can be noticed by testing.
10) Example: If you're testing for a drug and the LOD is 0.1 mg/mL, the test can tell that the drug is present at 0.1 mg/mL, but it can't give you the exact amount.
11) *LOQ (Limit of Quantification)*- LOQ is the lowest amount of a substance that can be measured accurately and precisely by the test. It's higher than the LOD and tells us the minimum amount that can be reliably counted or quantified.

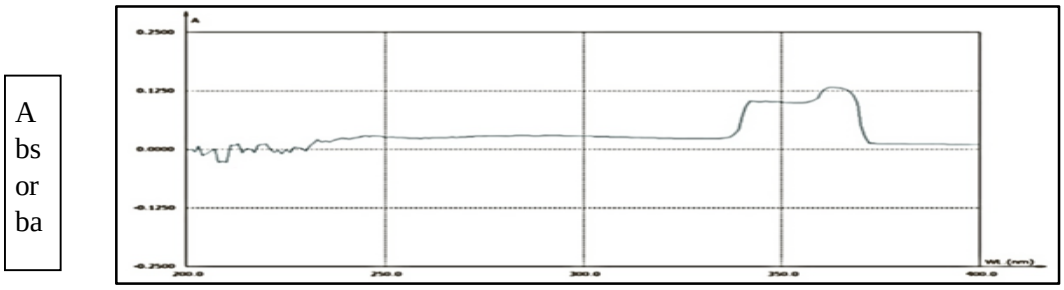
Example: If the LOQ is 0.3 mg/mL, then from this amount and above, the test can give you exact and trustworthy results.

III. MATERIALS & METHODS

1) *Materials*– The reference sample of Diclofenac Sodium was obtained from NOVARTIS INDIA LTD (Dr. Reddy). And the inactive ingredients were collected from the market which are used in the drug matrix.
2) *Diluent prepared*–Water and methanol in the ratio of 95:5, v/v is used as diluent in this technique.
3) *Standard preparation*–10 mg of diclofenac sodium was dissolved in 5 ml of methanol and mixed well. Then, 95 ml of water was added to make the total volume 100 ml, resulting in a 100ppm solution. From this, 5ml was taken and diluted to 50 ml using the same diluent.
4) *Test prepared*– 2 tablet were weighed of 594mg, that contain 200mg of diclofenac sodium and then powdered. Then the powdered sample equivalent to 100mg of diclofenac sodium was weighed and then placed into a 100ml of volumetric flask. 5ml of methanol was added and mixed well, after that, 95 ml of water was added to meet the volume up to 100ml. From this solution, 1ml was withdrawn and placed in a test tube . The volume was adjusted by the diluent up to 10ml and repeated this process for once.
5) *Instrument Used*–Single-beam UV Visible Spectrophotometer was used with matched quartz cell(1cm).

IV. RESULT AND DISCUSSION

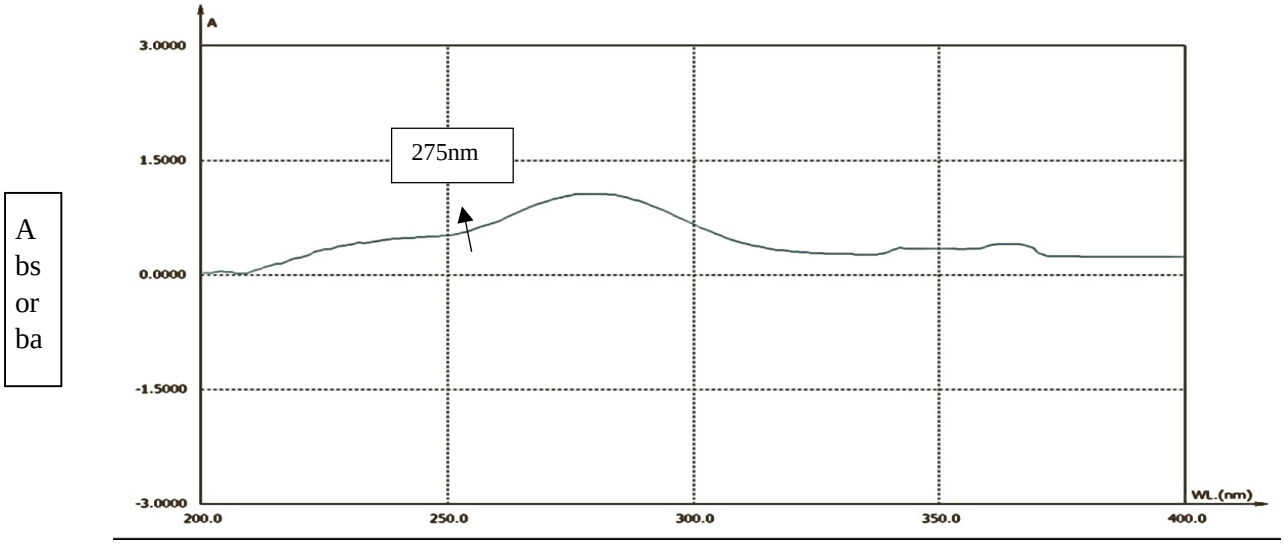
The solution of diclofenac sodium was scanned in UV Spectroscopy in b/w 200-400 nm. First using blank and then sample solution.



Absorba

The wavelength found to be 275nm Wavelength

Fig. 2. UV spectrum of blank preparation for diclofenac sodium



Wavelength

Fig. 3 UV spectrum for sample preparation

Validation of UV Spectroscopy for analysis of different parameters–

1) *Linearity:*Six points calibration curve were obtained in a concentration range from 0-10 ppm for Diclofenac sodium. The report of the drug was found to be linear in this investigation of concentration range VS absorption range.

Concentration	*Absorbance*
0	0
2	0.049
4	0.098
6	0.148
8	0.198
10	0.246

TABLE I Concentration VS Absorbance for Linearity Study

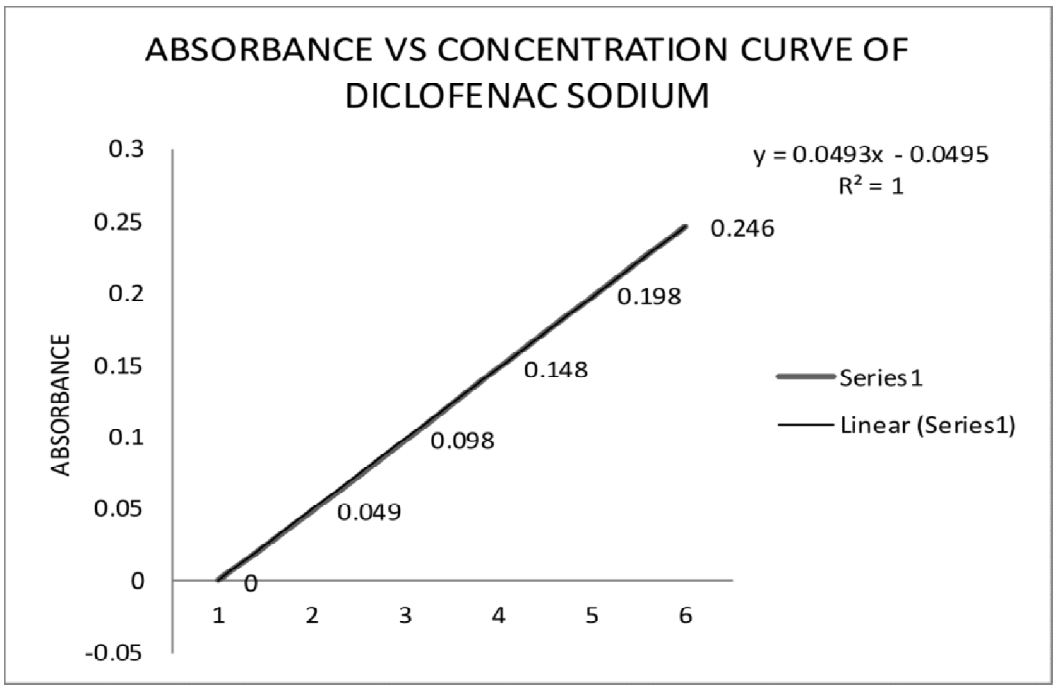


Fig. 4 Absorbance VS Concentration Curve

2) *Precision:* Precision of this analytical method is determined by conducting the analysis accordant with the procedure and following normal weight taken for analysis. Repeat the analysis six times. Calculate the % assay, mean assay, % Deviation and % relative standard deviation and %RSD of same concentrations.

Sample No.	Assay
1	100.8
2	100.2
3	100.3
4	100.5
5	100.6
6	99.9
Average	100.35
SD	0.35
%RSD	0.37960

TABLE II Repeatability Study of Precision

SAMPLE NO. (SET)	ASSAY	
	Intra-day	Inter-day
1	100.8	99.5
2	100.2	100.1
3	100.3	99.9
4	100.5	100
5	100.6	99.8
6	99.9	99.7
Mean	100.35	99.833
SD	0.35	0.19720
%RSD	0.37960	0.19753

TABLE III Evaluation Data of Intermediate Precision Study

3) *Accuracy:* Accuracy of the method is calculated by standard addition method at 3 stages. Standard quantity equivalent to 50%, 100% and 125% is to be added in the sample of diclofenac sodium. The result shown that best recoveries (98.86-99.32%) of the drug were obtained at each added concentration, those indicated that the method was accurate.

% Recovery level	% Recovery	Mean % Recovery	SD	%RSD
50%	98.86 98.85 98.84	98.85	0	0
			0.00816497	0.008279
100%	98.73 98.74 98.72	98.73	0.00693889	0.007041
			0.1503083	0.015351
125%	99.34 99.33 99.32	99.33	0.00707107	0.007134
			0	0

TABLE IV Evaluation Data of Accuracy Study

4) Robustness: Robustness target to the power of an analytical method to remain pure by small, deliberate variations in the parameters of the method—such as temperature, reagent concentration, pH or flow properties—so that it produces reliable results consistently.

Sr no..	Concentration	Absorbance		
		At 274nm	At 275nm	At 276nm
1	10µg/ml	0.247	0.254	0.258
2	10µg/ml	0.249	0.251	0.259
3	10µg/ml	0.249	0.248	0.261
4	10µg/ml	0.251	0.252	0.254
5	10µg/ml	0.254	0.249	0.251
6	10µg/ml	0.258	0.253	0.262
Average		0.251333	0.251167	0.2575
SD		0.003682	0.002115	0.003862
%RSD		1.464901992	0.841976	1.499887

TABLE V Evaluation Data of Robustness Study

V. CONCLUSIONS

The validated analytical method for the determination of diclofenac sodium in water was found to be accurate, precise. The method demonstrated good linearity in the range of tested concentration range. Recovery studies confirmed the accuracy of the method, while intra-day and inter-day precision results were within acceptable limits, indicating reproducibility. Overall, the validated method is reliable and suitable for routine analysis of diclofenac sodium in water samples, ensures compliance with environmental and pharmaceutical quality standards and cost effectiveness.

CONFLICTS OF INTEREST: Nil

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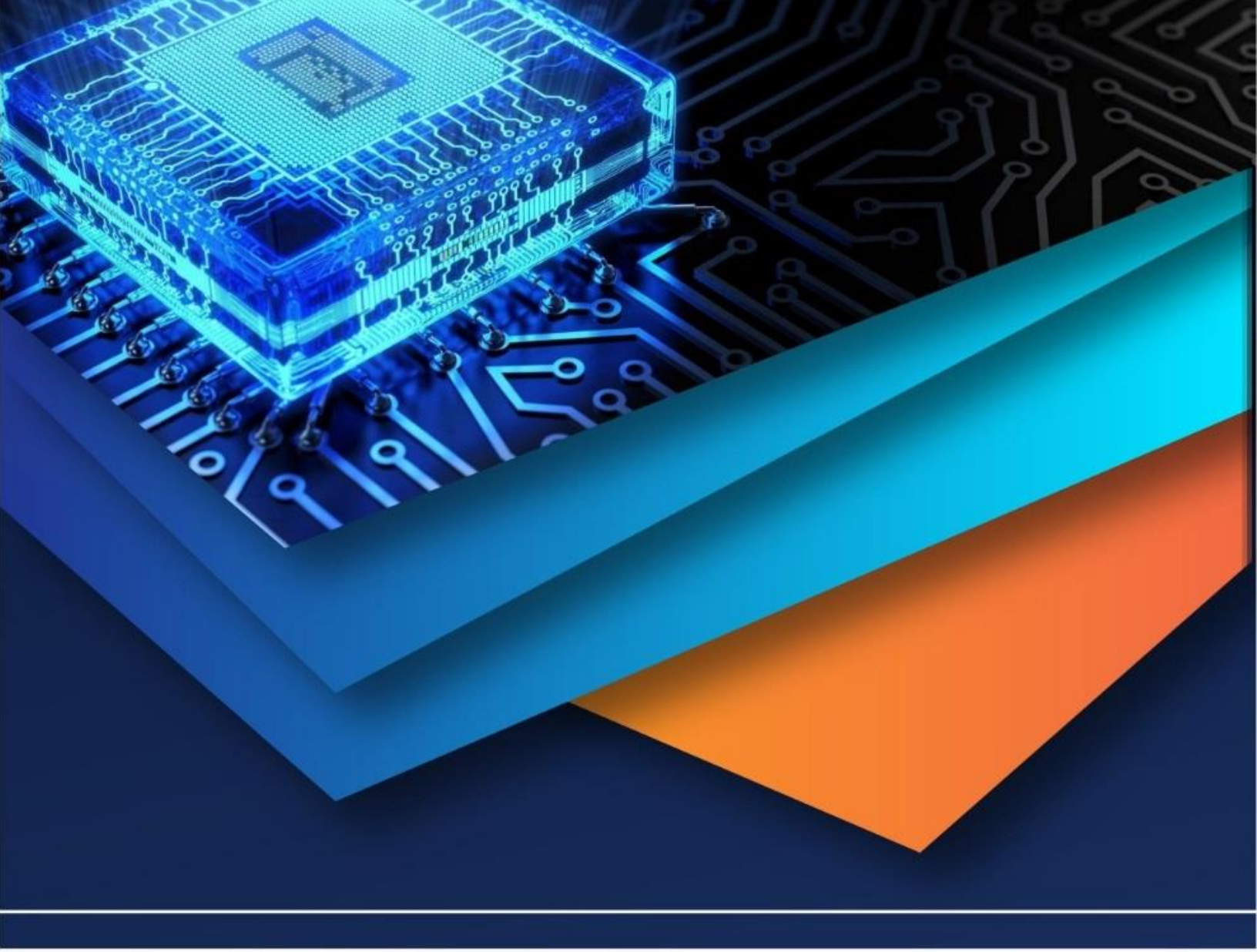

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