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Preparation and Characterization of Polycaprolactone Nanofibers for the Controlled Release of Chlorogenic Acid

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Abstract: ***Electrospun nanofibers have gathered more consideration in worldwide to deliver the bioactive agents in a sustained manner for biomedical applications. In this study, chlorogenic acid loaded polycaprolactone nanofibers were fabricated by electrospinning process and the average fiber diameters were analyzed using scanning electron microscopy analysis. Further, Fourier transform infrared spectroscopy results confirmed that the encapsulated chlorogenic acid was not modified chemically during the electrospinning process. In addition, sustained chlorogenic acid release profile was observed for 12 days. These results imply chlorogenic acid loaded polycaprolactone nanofibers have capability to deliver the chlorogenic acid in a sustained manner for various biomedical applications.***
Keywords: Electrospinning, Chlorogenic Acid, Polycaprolactone Nanofibers, Drug Delivery, Polymer

I. INTRODUCTION

In past few decades, electrospun nanofibers based drug delivery systems have gained more attention to improve the therapeutic efficiency in drug delivery applications [1], [2]. Electrospinning is an inexpensive and simple technique to synthesis the nanofibers from various natural and synthetic polymeric solutions [3]. Due to its high surface area-to-volume ratio, several bioactive agents (herbal extracts, genes and various hydrophilic and hydrophobic drugs) have delivered in a sustained manner for various therapeutic applications [1], [3]. In addition, the electrospinning process helps to produce the nanofibers with desired fiber diameters ranging from nano to micro [4]. Polycaprolactone is Food and Drug Administration approved semi-crystalline polymer has various advantages such as biodegradability, chemical stability, biocompatibility *etc* [1]. Several polycaprolactone based implants such as drug delivery sutures, fixation devices, wound dressings *etc* have used in various biomedical applications due to its slow bioresorption properties [5].

Chlorogenic acid, phenolic compound widely presented in fruits and vegetables such as pears, apple, lettuce, sweet potato, berries, coffee beans, spinach, tea *etc* [6]. It has various pharmacological properties such as antibacterial, antihypertensive, antitumor, anti-inflammatory, osteogenic potential *etc* [7]–[11]. Hence, sustained release of chlorogenic acid from polymeric nanofibers could enhance the therapeutic potential in various drug delivery and tissue engineering applications.

In this study, chlorogenic acid loaded polycaprolactone nanofibers were fabricated and characterized by scanning electron microscopy and Fourier transform infrared spectroscopy analysis. Further, sustained chlorogenic acid release profile was observed from chlorogenic acid loaded polycaprolactone nanofibers.

II. MATERIALS AND METHODS

A. Fabrication of Chlorogenic Acid Loaded Polycaprolactone Nanofibers

Chlorogenic acid loaded polycaprolactone nanofibers was fabricated using ESPIN – NANO (PECO – Chennai, India) [12]. Chlorogenic acid (Himedia, India) was added at the concentration of 3mg/ml to the prepared 12% (w/v) polycaprolactone (Sigma-Aldrich (St. Louis, MO), average Mn 80,000) in 1:1 of dichloromethane and dimethylformamide solution. Further, the prepared chlorogenic acid mixed solution was transferred into a syringe fitted with the needle (0.45×13 mm) and electrospun with a 15 kV applied high-voltage, collector drum speed of 1500 rpm, 2 ml/hr flow rate and 15 cm needle tip to collector distance. Similarly, the polycaprolactone nanofibers were synthesized without chlorogenic acid was considered as control.

B. Characterization of the Fabricated Nanofibers

1) *Scanning Electron Microscopy (SEM):* The morphology of the polycaprolactone nanofibers and chlorogenic acid loaded polycaprolactone nanofibers were evaluated by using SEM (TESCAN VEGA3 SBU) analysis. For this, the prepared nanofibers were cut into small pieces and sputtered with gold; analyzed with a 10 kV of applied voltage and 5000x magnifications. The

average fiber diameters were calculated manually from randomly selected 20 fibers using ImageJ software (ImageJ 1.51j8, National Institutes of Health, USA). The average fiber diameters values are expressed as mean ± standard deviation.

2) *Fourier Transform Infrared Spectroscopy (FTIR):* Polycaprolactone nanofibers, chlorogenic acid and chlorogenic acid loaded polycaprolactone nanofibers were mixed with potassium bromide separately and the pellets were prepared. The prepared pellets were analyzed by FTIR spectrophotometer, operating at 4,000–400 cm^{-1} with 1cm^{-1} resolution.
3) *Chlorogenic Acid Release from Chlorogenic Acid Loaded Polycaprolactone Nanofibers:* Chlorogenic acid loaded polycaprolactone nanofibers (1cm×1cm) were incubated in 2ml of Dulbecco's phosphate buffered saline (DPBS). Further, 700µl were collected at fixed time interval and replaced with the same volume of DPBS. Furthermore, collected samples were read at 324 nm using UV-Vis spectrophotometer and the released chlorogenic acid concentrations were calculated from the chlorogenic acid standard curve.

III. RESULTS AND DISCUSSION

Chlorogenic acid was successfully incorporated into polycaprolactone nanofibers and the morphology of the fabricated nanofibers was evaluated using SEM analysis. The results (Fig. 1) showed that the prepared fibers were smooth and the average fiber diameters were found as 549.68±195.95nm and 623.50±244.94nm for both polycaprolactone nanofibers and chlorogenic acid loaded polycaprolactone nanofibers respectively. Previous studies have reported that drug loaded polymeric nanofibers has showed an increased average fiber diameters compared to control [1], [12]–[14]. Thus, this SEM results suggested that an increased average fiber diameters were observed in chlorogenic acid loaded polycaprolactone nanofibers compared with polycaprolactone nanofibers.

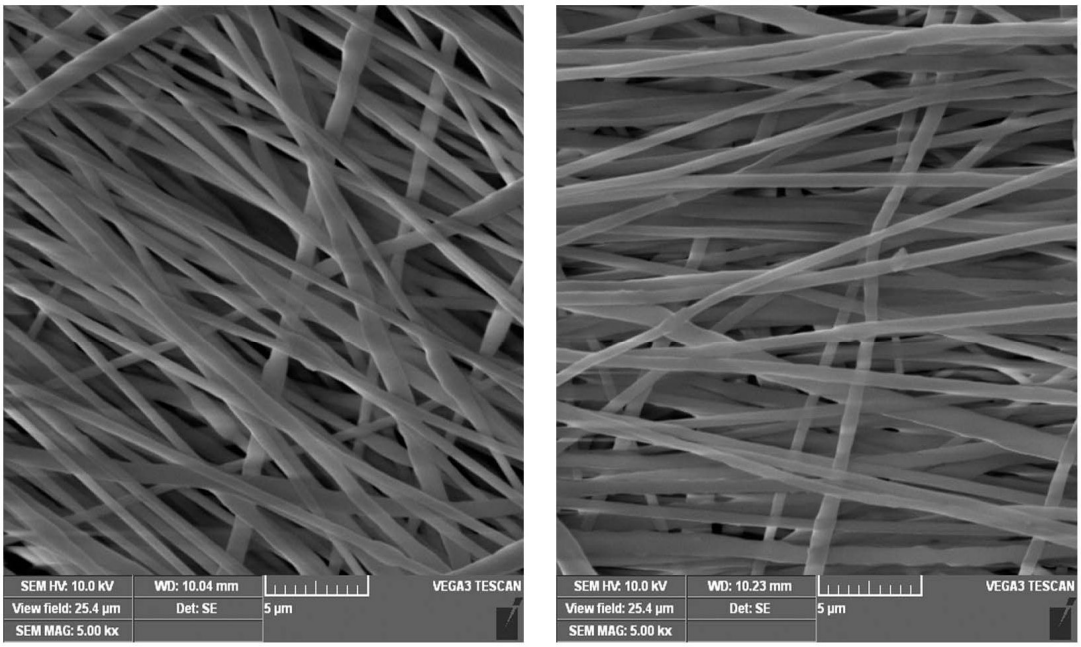

Fig. 1 SEM images of prepared nanofibers. Where, a - Polycaprolactone nanofibers; b - Chlorogenic acid loaded polycaprolactone nanofibers

Further, FTIR spectra showed the characteristic peaks of polycaprolactone nanofibers (Fig. 2A), chlorogenic acid (Fig. 2B) and chlorogenic acid loaded polycaprolactone nanofibers (Fig. 2C). The characteristic peaks of polycaprolactone was observed at 2935.9 cm^{-1} (symmetric CH2- stretching) and 1235.8 cm^{-1} for polycaprolactone nanofibers [15], [16]. The bands were observed at 2953 cm^{-1}, 1687 cm^{-1}, 1639.5 cm^{-1}, 1603.1 cm^{-1}, 1442.7 cm^{-1}, 1289.1 cm^{-1}, 1113.8 cm^{-1}, 977.2 cm^{-1}, 818.3 cm^{-1} and 603.1 cm^{-1} represents the chlorogenic acid [17]. For chlorogenic acid loaded polycaprolactone nanofibers, the bands at 2942.8 cm^{-1}, 2864.2 cm^{-1} (asymmetric CH2- stretching) and 1722.3 cm^{-1} indicating the presence of polycaprolactone [15] and the bands at 1447.7 cm^{-1}, 1108.7 cm^{-1} and 818.2 cm^{-1} which confirmed the presence of chlorogenic acid [17]. The FTIR results suggested that the chlorogenic acid was not undergone any chemical modification after the encapsulation process.

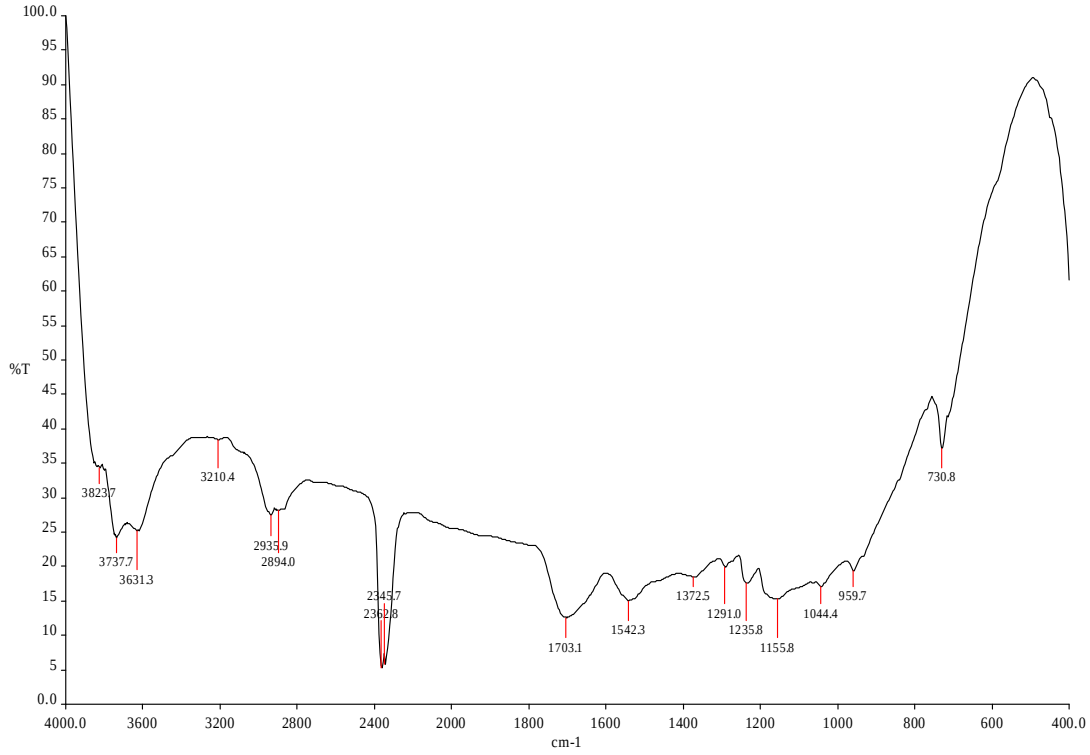

Fig. 2A FTIR spectra of polycaprolactone nanofibers

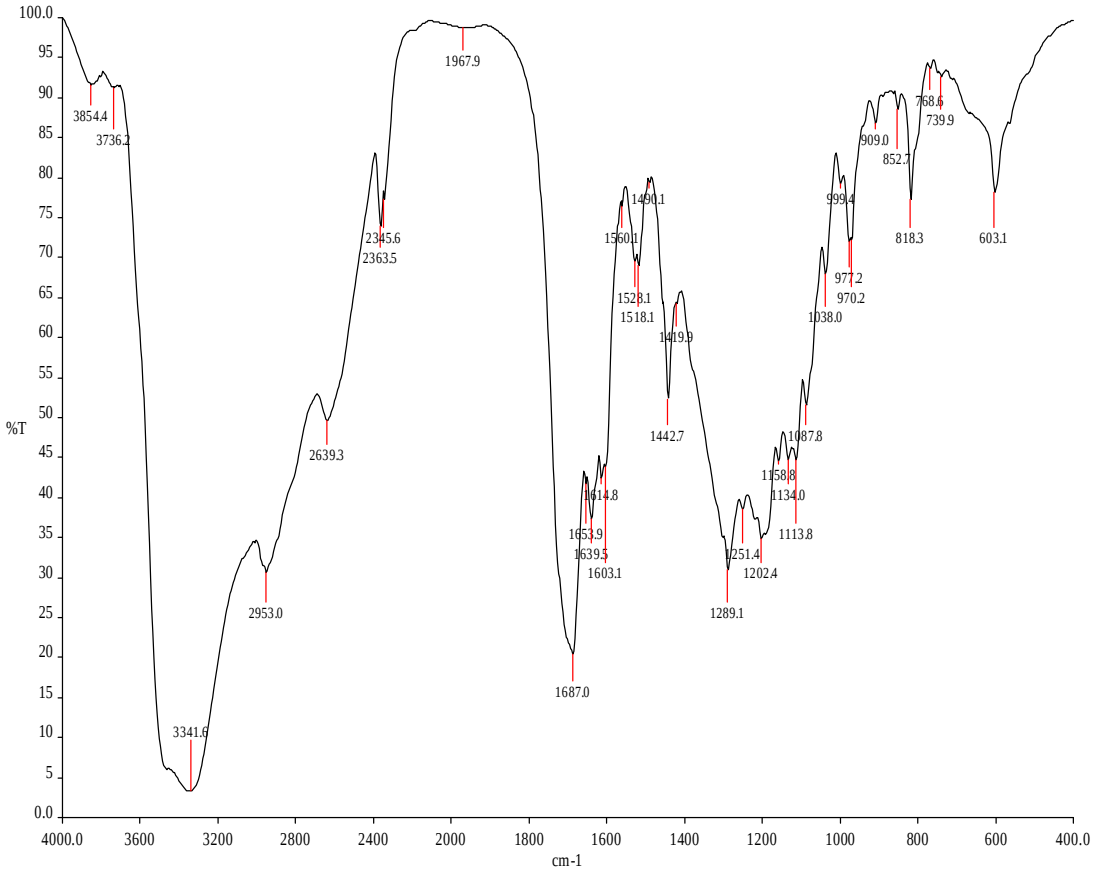

Fig. 2B FTIR spectra of chlorogenic acid

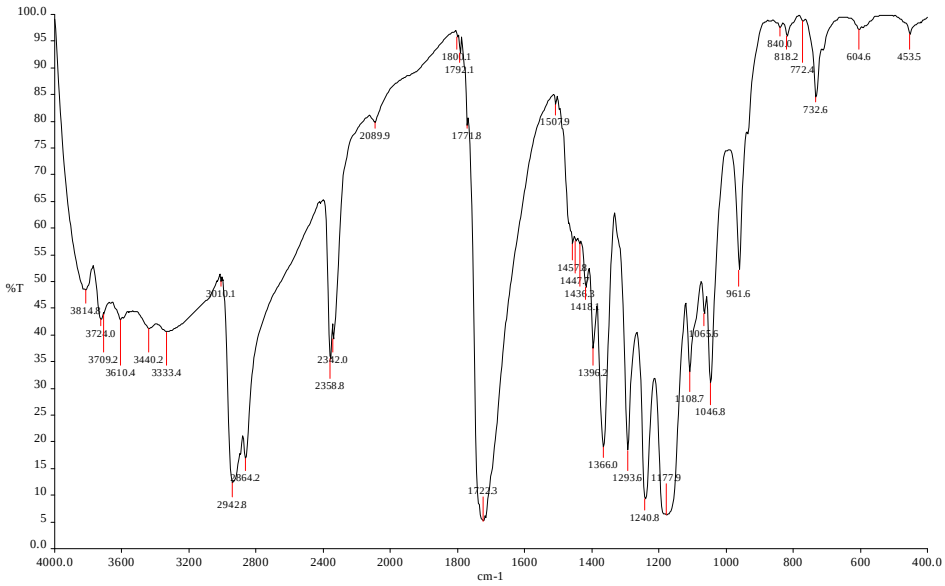

Fig. 2C FTIR spectra of chlorogenic acid loaded polycaprolactone nanofibers

The sustained release of chlorogenic acid was observed and the cumulative release concentration was found as 50.9 micromolar (µM) on day 12 (Fig. 3). Several studies have reported on delivery of various bioactive molecules in sustained manner from a wide range of polymeric nanofibers [1], [18]–[21]. In addition, the polymeric nanofibers could support to encapsulate and also enhances the dissolution of the water insoluble drugs due to its high surface area to volume ratio properties [22], [23]. Furthermore, the drugs chemical integrity properties were not altered by the electrospinning process [23], [24]. Thus, the chlorogenic acid release profile suggested that the chlorogenic acid was successfully released in a sustained manner from the chlorogenic acid loaded polycaprolactone nanofibers which could facilitate for an effective therapeutic potential in various drug delivery and tissue engineering applications.

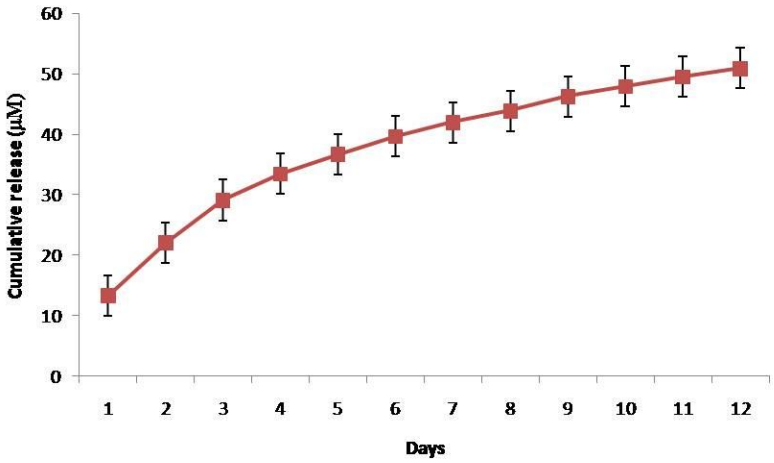

Fig. 3 Cumulative release of chlorogenic acid from chlorogenic acid loaded polycaprolactone nanofibers

IV. CONCLUSION

Chlorogenic acid was effectively encapsulated into polycaprolactone nanofibers by electrospinning process. The prepared chlorogenic acid loaded polycaprolactone nanofibers have showed an increased average fiber diameters compared to control. Also, the FTIR results suggested that there was no chemical modification of chlorogenic acid during the encapsulation process. In addition, chlorogenic acid loaded polycaprolactone nanofibers showed the sustained chlorogenic acid release profile for 12 days. These findings conclude that the fabricated polycaprolactone nanofibers have capability to deliver the encapsulated chlorogenic acid in a sustained manner for various drug delivery and tissue engineering applications. Furthermore, the effect of various

electrospinning parameters on chlorogenic acid loaded polycaprolactone nanofibers need to study in detail to attain the preferred average fiber diameter as well as chlorogenic acid release profile for tissue engineering applications.

V. ACKNOWLEDGEMENT

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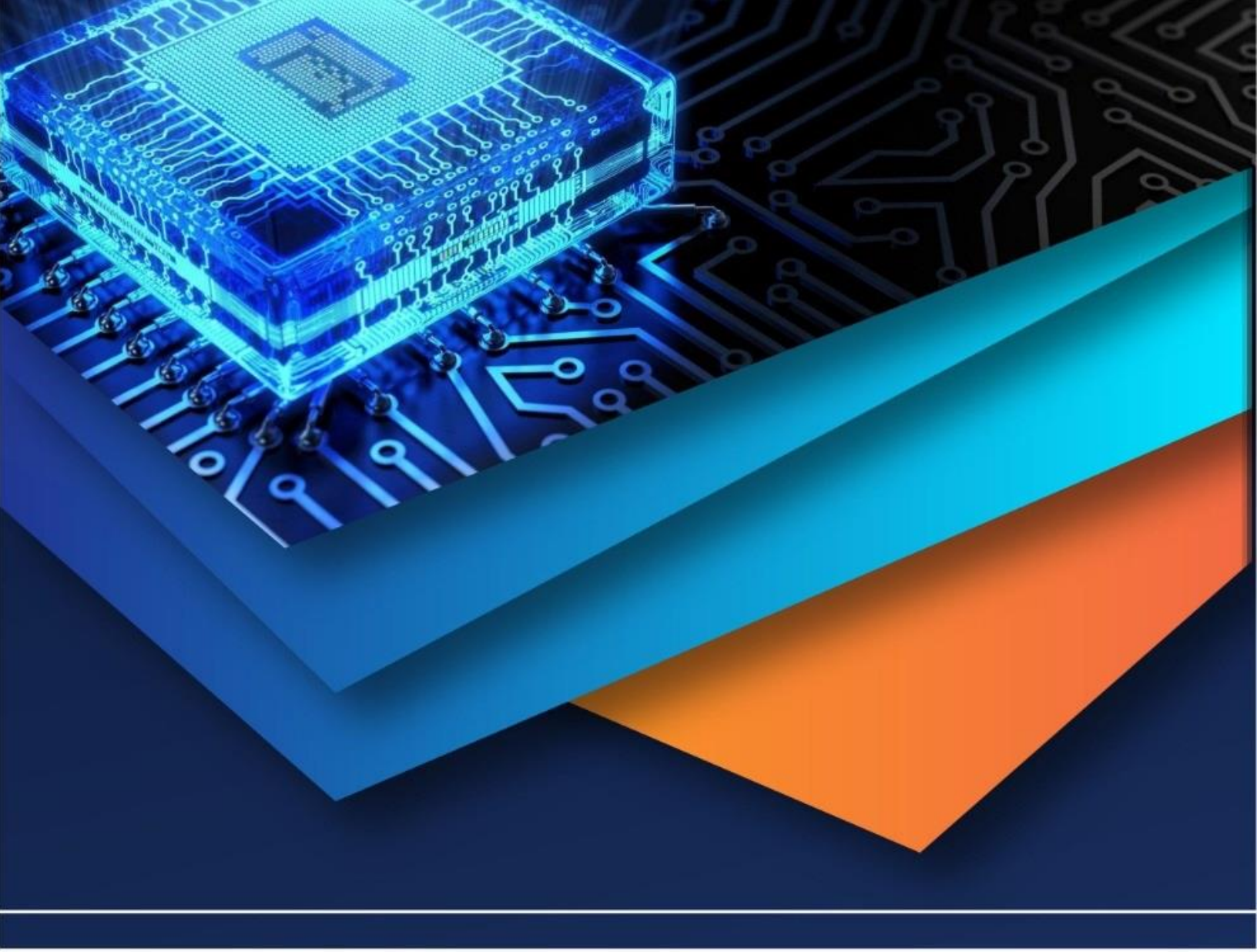

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