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Bio-Mass Material as a Source of Micro-crystalline Cellulose by Strong Acid Treatments.

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Abstract: ***Due to environmental concerns agro-waste material namely wheat straw (WS) will be utilized to produce microcrystalline cellulose (MCC) via mercerization, bleaching and strong acid hydrolysis. The current investigation compared MCC produced by phosphoric acid (H3NPO4), sulfuric acid (H2SO4) and hydrochloric acid (HCL). The percentage yield of each process were compared and found to be the highest for the H3PO4 compared H2SO4 and HCL. The morphology of the three products was viewed under scanning electron microscope (SEM). The heat of dissolution was studied for the three samples, and found to be the highest for the H3PO4, which is in line with %yield. This is also indicative that the chemical attack on the amorphous regions was the highest for H3PO4.***

I. INTRODUCTION

Manufacturing of high performance engineering materials such as micro crystalline cellulose (MCC) from renewable resources such as agro-waste materials is one of ambitious goals currently being pursued by researchers all over the world. MCC is a biodegradable material which is of porous particle texture. The MCC find its way to applied in industries such as pharmaceutical, cosmetics and food industries as well as water retainer and reinforcement filler in bio-composite fabrication.

Due to the increasing concern about the environment and CO_2 emissions, the scientific community have considered the agricultural wastes such as coconut husk fibers, groundnut husk, rice straw and rice husk to produce micro crystalline cellulose (1-5). The choice of the agro-waste is related to geographical regions, in other words to what is common and abundant in the region. Referring to agro-waste materials in Jordan, wheat straw (WS) and olive pomace (OP) are two common agricultural wastes they were used as building material, burned in the open atmosphere which produces a lot of CO_2, further it is being used to feed animals especially the wheat straw. However, recently various attempts were made by us to produce MCC from olive pomace (6-18), but up to our knowledge no attempt has been made to extract MCC from the WS and. In this work MCC will be extracted from wheat straw (WS) by the removal of non-cellulosic components relying on mercerization treatment of the WS followed by hydrolysis technique.

II. EXPERIMENTAL

A. Materials

WS was collected from a local wheat farm in Jordan. Sodium hypochlorite (NaOCl) and sodium hydroxide (NaOH) were used as bleaching agents while H_3PO_4, H_2SO_4 and HCl were used for acid hydrolysis process. All chemical were used without further purification.

B. Alkaline and Bleaching Treatments of WS

The received WS was washed with distilled water and dried under air for fourteen days followed by drying using air circulating at 100 °C. The dried sample was ball milled followed by sieving on a Retsch AS 200 shaker for ten minutes. WS powder was treated with 10% aqueous sodium hydroxide (NaOH) solution at room temperature, maintaining a liquor ratio of 20:1 for 24h to remove the hemicelluloses and other greasy materials. The solution was filtered and the residue washed with distilled water for removal of solubilized components until pH 7 was achieved. The alkaline treated WS then was undergone bleaching treatment with 5% NaOCl for 6h at room temperature. The resulted cellulose obtained from the treated WS was filtered and rinsed several times using distilled water. It was dried in an oven until constant weight was recorded. The dried powder was grounded into fine powder using rotary ball mill (19-22).

C. Acid Hydrolysis

The obtained cellulose was hydrolyzed using 5% acid solution at room temperature under continuous stirring. The MCC collected was filtered and rinsed with distilled water until pH 7 was achieved followed by air circulating oven drying until constant weight was obtained. The % yield of the MCC calculated according to equation 1 (16) is reported in table 1.

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$$x = \frac{B}{A} * 100 \quad (1)$$

Where B is the final weight in grams and A is the initial weight.

III. RESULTS AND DISCUSSIONS

A. Percentage Yield of MCC

The effect of acid hydrolysis on the % yield of WS is reflected in Table 1.

ingredient(g)	W1(g)	W2(g)	%Yield
Ethanol	100	90	90
Sodium hydroxide	90	70	78
Hypochlorite	70	60	86
Hydrochloric acid	20	15	75
sulfuric acid	20	13	65
phosphoric acid	20	14	70

Table1: percentage yield of MCC extracted by various strong acids treatments.

In this regard the mercerized samples have been exposed to three different acids. These were H_3PO_4, H_2SO_4 and HCl at 5% concentration. The rationale behind the acid treatment is based on the fact that the hydronium ions of the mineral acids are capable to penetrate and remove the excessive amorphous regions of cellulose; this fact was adopted and used to explain the obtained results in this study. Note that the amount of MCC obtained after the HCl treatment was the highest followed by H_3PO_4, H_2SO_4 respectively. The different yield% should be correlated to the solvating power of each acid. It is noted that sulfuric acid treatment has come out with the less amount of % yield; this is a hint that the sulfuric acid has the highest solvating power toward the WS compared to the other two acids. This is a remark that the sulfuric acid was able to access the WS and to dissolve higher amounts of the amorphous regions within the WS; hence lower % yield was detected but more crystalline product. To further support this tend the heat of dissolution was estimated using bomb according to equation 2. The results of the calorimetry study is shown in table 2.

$$Q = cm\Delta T \quad (2)$$

Where: Q is the heat released, c, is the specific heat, m is the mass, and ΔT is the change in temperature. Note that the heat of dissolution (Q) is the highest in case of sulfuric acid treatments compared to the other two acids. This is again confirms our claim that the sulfuric acid has the highest solvation power, in other words it has dissolved the maximum amount of amorphous cellulose, consequently the least %yield.

Chemical Agent	Q1 (kj/g)	Q2(kj/g)	ΔQ(kj/g)
HCl	3	2	1
H_3PO_4	8	4	4
H_2SO_4	51	34	17

Table 2: shows the heat of enthalpy for the reactions with the wheat straw

B. Scanning Electron Microscope Study

Figure 1 shows the SEM micrographs of the raw and hydrolyzed biomass. The microstructure of these samples was examined to understand the influence of chemical treatment on the structural changes, particle size and shape of the biomass powder. It can be seen that the raw material (as received) is a non-porous solid mass.

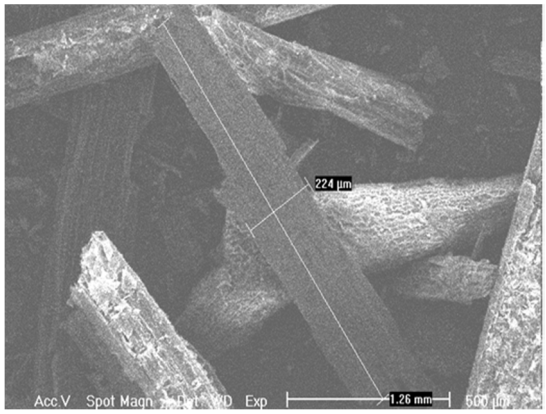

Fig.1: SEM images of the as received WS.

Sample	Width (µm)
Raw sample	224
HCl treated	31.5
H_3PO_4 Treated	42.5
H_2SO_4 Treated	41.5

Table3: The effect of various acid treatments on the particle size of the WS.

Furthermore, it is noted that the material is not isometric with fiber like or agglomerates bundled together with high dimeter as evidenced from the scanning electron microscope images displayed in Figure 1. In this regard the influence of the acid treatment on the particle size of the WS is compared in table 3. In term of particle dimensions, it is noted that external fiber diameter (width) is 224 µm. The effect of acid treatment on the alkali bleached WS sample is show in in Figures 2-4 consequently. Figure 2 displayed the microstructure of the hydrochloric acid treated sample. The aim is to compare its potential to release the crystalline regions with other acid potentials.

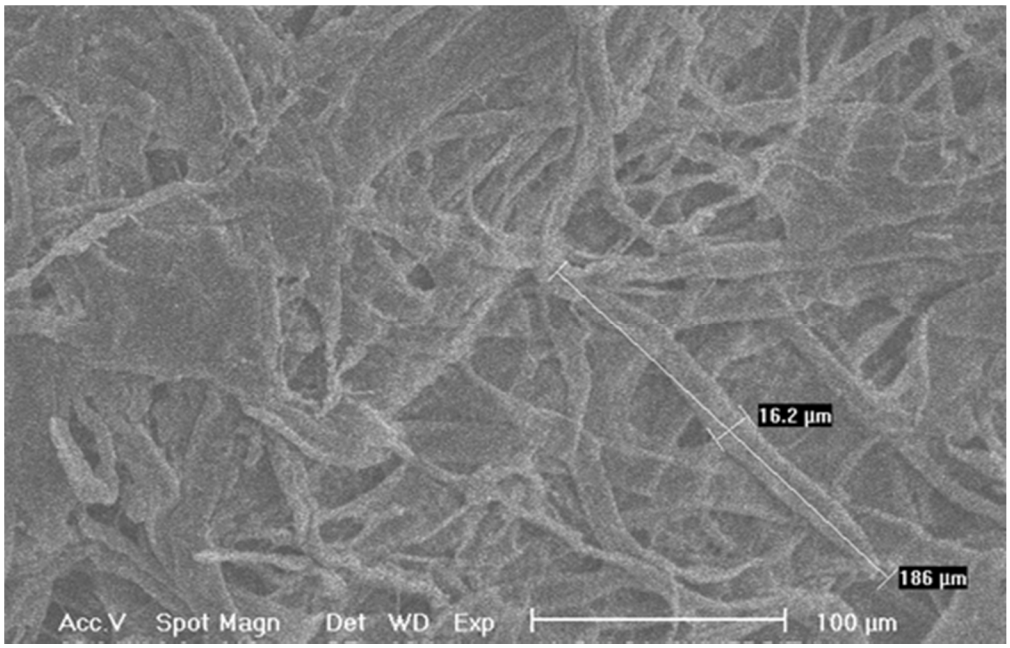

Fig.3: SEM images of the Phosphoric acid hydrolyzed WS.

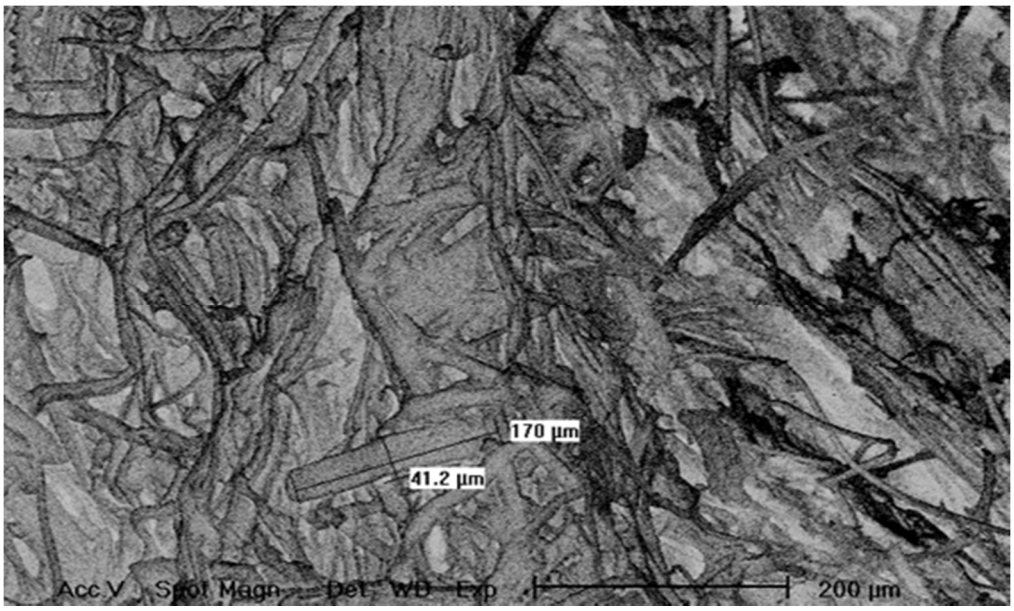

Fig.4: SEM images of the sulfuric acid hydrolyzed WS.

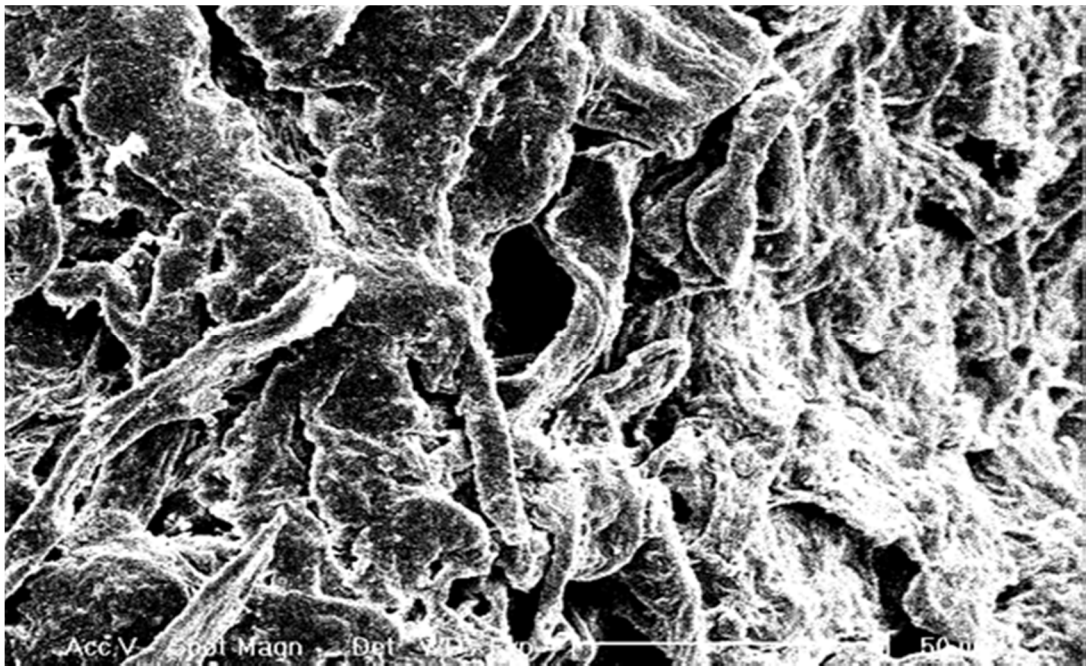

Fig.5 : SEM images of the hydrochloric acid hydrolyzed WS.

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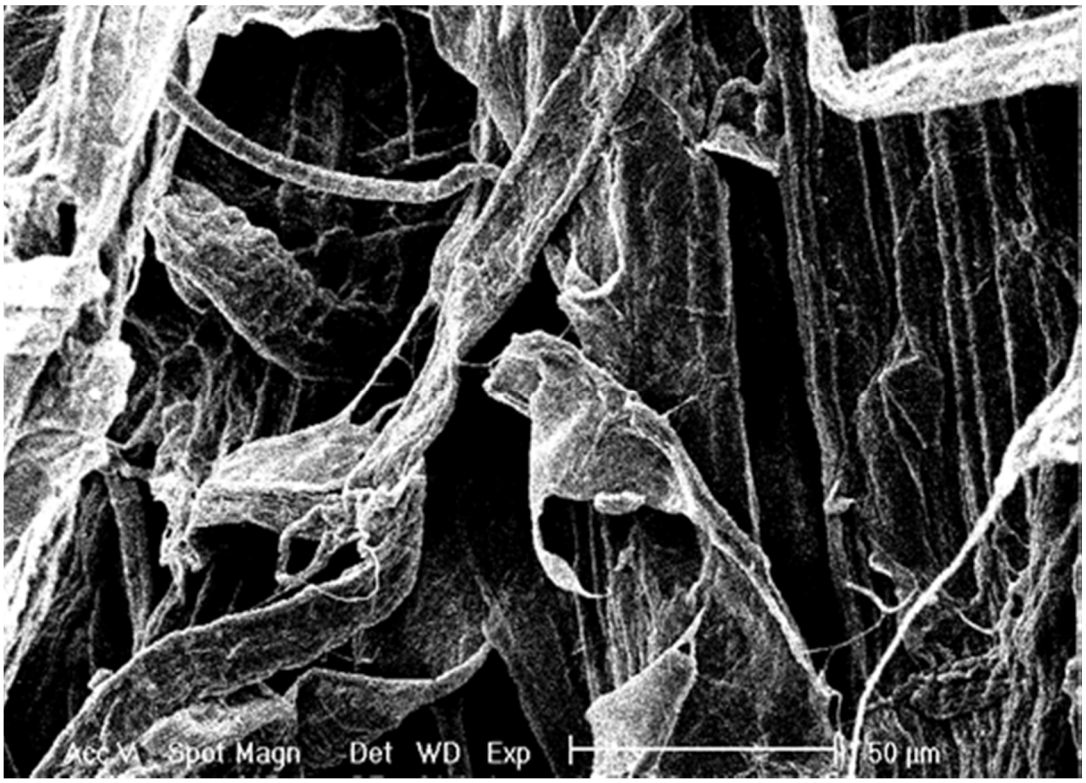

Fig.6: SEM images of the phosphoric acid hydrolyzed WS

IV. CONCLUSION

Based on the aforementioned results, the following facts can be concluded

A. The alkali treatment followed by acid hydrolysis was able to dissolve the non-cellulosic components and to release the MCC-WS.

B. The sulfuric acid based hydrolysis displayed the best results compared to the other two acidic treatments. This was evidenced by the higher aspect ratio of the MCC shown in table3 and the improved surface area shown by SEM images.

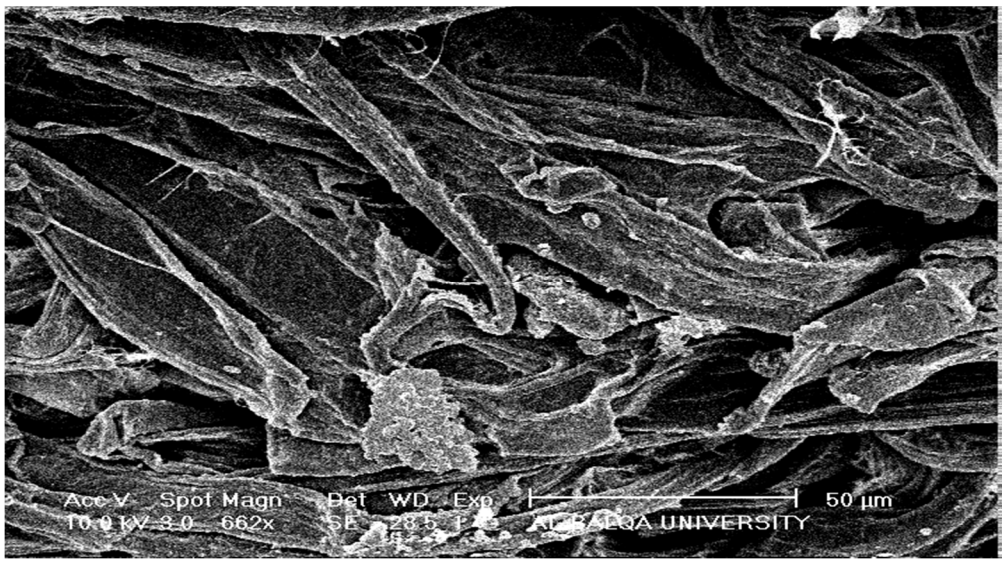

Fig.7: SEM images of the sulfuric acid hydrolyzed WS.

It is clear that the hydrochloric acid treatment has tremendously influenced the surface topography. Note that surface texture has been converted to thinner fiber like material as indicated by the lower dimeter (31.5µm). On the other hand the phosphoric acid treated material shown in Figure 3 showed further improvements in the sense that more dimensional reduction has occurred compared to that of hydrochloric acid treatments. This should be attributed to the solvating power of the acid and its capability to extract the amorphous regions. Figure 4 demonstrates the influence of sulfuric acid treatment on the microstructure of the WS compared to the other acidic treatments. It is clear that further reduction in the dimension were obtained, this is again should be related to the removal of the amorphous regions. As a result of the particle size reduction due to the breakdown of the cellulose molecules, one can expect that such dimensional change was accompanied by an increase in the surface area of the treated WS materials, remember that that the smaller the particle size the higher the surface area is. Furthermore, the enhanced surface area could be indirectly quantified from the SEM micrographs as shown in 5-7, consequently. Apparently the surface appearance was converted to flattened shape with respect to the raw sample shown in Figure 1.

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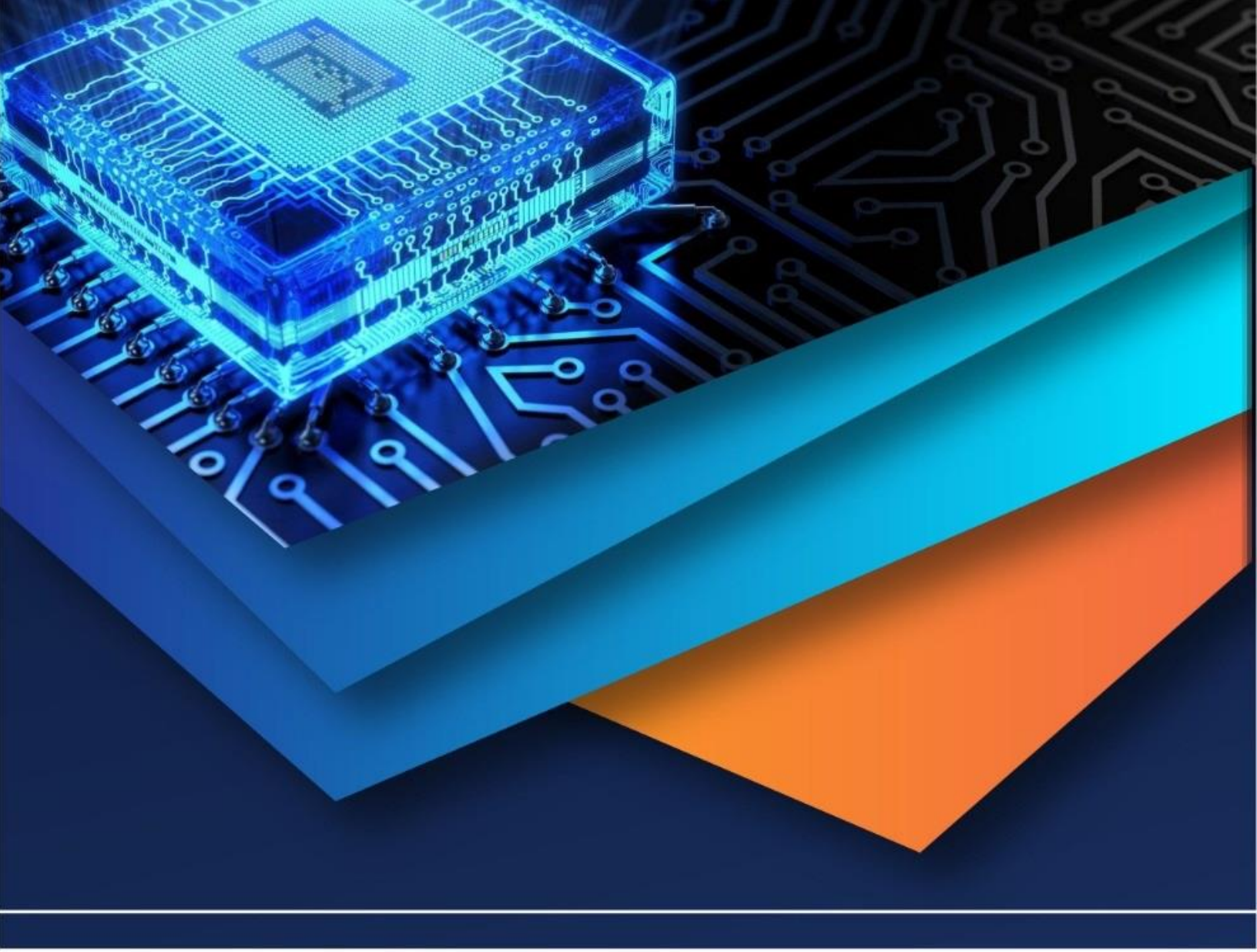

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