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Synthesis of Furfural from Corn Cobs by Using Membrane Technology

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Abstract -Today all major companies used furfural as a selective solvent in refining of lubricating oil. Furfural is produced from agricultural waste biomass that contain pentosans, which are aldose sugars, composed of small rings formed from short five-member chains, that constitute a class of complex carbohydrates, present in cellulose of many woody plants such as corn cobs, sugar cane bagasse, rice and oat hulls etc. The pervaporation performance of the membranes studied with aqueous furfural solution as feed. The effects of feed composition and temperature on the membrane performance also studied. (Keywords- Corn cobs, Hydrolysis, PV, Furfural)

I. INTRODUCTION

India has agro-based economy and agriculture accounts for a large share of GDP and employment. Besides, a number of useful products, a large quantity of agro-wastes are also produced which is not properly used at present. These agro-wastes are either burnt as fuel or improperly disposed off causing the problem of waste treatment and environmental pollution. In the recent years several attempts have been made to prepare furfural from cheap raw materials like Corn cobs, Sawdust, Rice husk, Sunflower husk, Cottonseed hulls and Bagasse. In continuation of previous attempts, present work is planned to identify and evaluate different bio raw material for the production of furfural economically on industrial scale. Furfural1 is produced from agricultural waste biomass that contain pentosans, which are aldose2 sugars, composed of small rings formed from short five-member chains, that constitute a class of complex carbohydrates, present in cellulose of many woody plants such as corn cobs, sugar cane bagasse, rice and oat hulls etc. Furfural is a clear, colorless motile liquid with a characteristic 'almond-benzaldehyde' odor. The molecular formula is C5H4O2. Its synonyms are: 2-furancarboxaldehyde, furaldehyde, 2-furanaldehyde, 2-furfuraldehyde, fural, furfurol. When exposed to sunlight in the presence of oxygen auto-oxidation occurs and it darkens to a dark red/brown color. In theory, any material containing pentosans can be used for the production of furfural. Technically furfural is produced by acid hydrolysis of the pentosan contained in woody biomass. Membrane separation is a potential technique for separation of toxic chemicals from aqueous streams. It includes micro-filtration, ultrafiltration, Reverse osmosis and pervaporation. Pervaporation (PV) is considered to be a promising alternative to conventional energy intensive technologies like extractive or a zeo tropic distillation for the separation of components of liquid mixtures for being economical, safe and ecofriendly. The separation of compounds using pervaporation technique can be classified into three major fields, viz. (i) dehydration of aqueous– organic mixtures (ii) removal of trace volatile organic compounds from aqueous solutionand (iii) separation of organic–organic solvent mixtures. Separation of furfural from aqueous solution is in the second category. Recently, we have reported the pervaporative separation of furfural from aqueous solution by hydrophobic micro porous PTFE (polytetrafluoroethylene) membrane and have found the membrane to be furfural selective. The performance of the membrane was monitored with the variation in porosity of the prepared membrane. Moreover, the influences of operating conditions such as feed concentration and temperature on the separationperformance of the membranes were studied. The application of furfural are as it is used as selective solvent in the refining of lubricating oils, Its derivative Furfuryl alcohol is used as motor fuel and preservative for biological specimen due to its germicidal character, It is also used as decolorizing agent, It is also used in extraction of isoprene from other c4 and c5 hydrocarbons, It is also used to produced resins, For extraction of butadiene from cracked refinery gases, Furfuryl alcohol is used in hypergolic propellant which are used as fuel in rocket. There are numbers of study have done on the synthesis of Furfural, carried out different methods and got different % yield. But from these works yet the % yield not got more than 60% of maximum theoretical yield. So we need good separation technology for getting more % yield. In this work we uses the membrane technology for the maximum % yield optimization. By this study demonstrates that membrane technology can serve as a highly selective, cost & energy saving technology. We also study the different parameters like as effect of feed composition on permeate concentration of furfural, effect of temperature on flux of furfural etc.

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II. MATERIALS AND METHODOLOGY

Corn cobs used for the synthesis of furfural which are contains

Cellulose → 32.3 - 45.6%

Hemicelluloses → 39.8%

Lignin → 6.7 – 13%

Corn cobs have been used on a small scale as a fuel for direct combustion in cooking and heating. Corn cobs are not harvested, stored, or utilized in a moisture free condition. Use of corn cobs as a biomass feedstock offers promising possibilities for renewable energy production. Corn cobs have a bulk density of about pounds per cubic foot and may be stored economically in the open in a cheap wire-fenced enclosure. Cobs are of little commercial value, but because the gain is generally shelled at individual farms or certainly in scattered rural areas, collection and transportation of cobs to the manufacturing plant becomes a major consideration of cost. Some 12,000tons per annum of cobs would be required to produce 1,000tons of furfural per annum. It would seem therefore that corn cobs alone would never entirely fulfill raw material demands. The methodology of synthesis of furfural is contains Pretreatment Process, Hydrolysis, Separation Processes, Purification Processes

A. Pretreatment Process

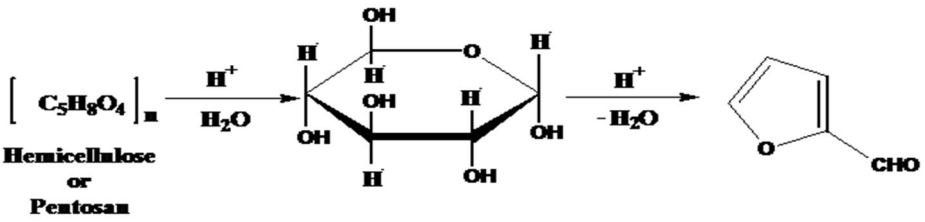
In this process we crushed the corn cobs into 1mm-10mm sized chips and dried it up to 110°C





B. Hydrolysis Process

In the process of hydrolysis we used 500 grams crushed corn cobs as a raw material which hydrolysed by using H_2SO_4 acid with differentconcentration16% / 14%). Digestion time for hydrolysis process is nearly about 100min in which NaCl used as catalyst. Experiment is carried out in simple distillation set up. Then distillate is taken to separation process.



Pentosan is one of the important fibre components of non-starch polysaccharides called hemi-cellulose. Hemicellulose can be any of several hetropolymers present in almost all cell walls.

C. Separation And Purification Process For Furfural

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After hydrolysis we have to separate furfural from hydrolyzed solution therefore we used the Pervaporation process for separation of furfural. Pervaporation is the combination of evaporation and permeation. In this process we have used PV apparatus in which PTFE (polytetrafluoroethylene) membrane of 0.2µm pore diameter and the cross section area is found 5.06cm^2.



The hydrolyzed solution fed to the flask of PV apparatus then temperature is increased up to 50-80°C and then vaccume pump is started and permeation of furfural is carried out2mm.hg.because PTFE is Hydrophobic membrane which is only organophilic. This operation is carried out upto 30min -1hr. After permeation we condensed the Vapour of Furfural and we get product furfural in pure form.

D. Method Used and Concentration For Conformation Determination Analysis Of Product

Absorption Spectroscopic methods of analysis based upon the fact that compounds ABSORB light radiation of a specific wavelength. In the analysis, the amount of light radiation absorbed by a sample is measured. The light absorption is directly related to the concentration of the coloured compound in the sample. Here we were taken different standard concentrations of furfural as like as 2, 4, 6, 8,…....,up to 100% and measured the absorbance at constant 340nm wavelength for different concentrations.

III. RESULT AND DISCUSSION

A. Hydrolysis Of Corncobs

Sr.no.	Weight of corncobs in gram	Hydrolysing acid with concentration	Temperature for hydrolysis	Concentration of furfural estimated in hydrolysed solution
1	500	14% H_2SO_4	120°C	8.8%
2	500	14% H_2SO_4	120°C	8.5%
3	500	14% H_2SO_4	120°C	8.2%
4	500	16% H_2SO_4	120°C	9%
5	500	16% H_2SO_4	120°C	9.2%
6	500	16% H_2SO_4	120°C	8.9%

Observation table no.1- for process of Hydrolysis

B. Pervaporation Of Hydrolyzed Solution

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Sr.no.	Volume of hydrolysed solution in ml	Concentration of hydrolysed solution	Volume of pervaporated in ml	Concentration of furfural in pervaporated solution
1	200	8.8%	56	54%
2	200	8.5%	53	57%
3	200	8.2%	50	55%
4	200	9%	58	59%
5	200	9.2%	59	56%
6	200	8.9%	54	57%

Observation table 2- for pervaporation of hydrolyzed solution

C. Distillation Of Pervaporated Solution

Sr. no.	Volume of permeate taken in ml	Volume of distillate in ml	Volume of residue in ml	Concentration of furfural in residue
1	56	27	29	97%
2	53	21	32	95%
3	50	20	30	92%
4	58	25	33	99%
5	59	25	34	94%
6	54	22	32	98%

Observation Table no.3- for distillation of pervaporated solution

In above process we have used 500grams of corncob as a raw material in each time as shown in table no.1. The temperature for hydrolysis was also taken constant as 120°C and time for hydrolysis was also same for each batch as100minute.In above process we used two different concentrations of sulphuric acid was taken as 14% and 16%. For 16% acid concentration we get maximum conversion of Pentoses to furfural. The hydrolysis process was carried out in simple distillation set up; in distillate we get hydrolyzed solution which contained mixture of water, furfural and some light fractions like as methanol acetone etc.

After hydrolysis we have to separate furfural from hydrolysed solution therefore we used the Pervaporation process for separation of furfural. In this process we have used PV apparatus in which PTFE (polytetrafluoroethylene) membrane of 0.2µm pore diameter and the cross section area is found 5.06cm^2. The hydrolysed solution fed to the flask of PV apparatus then temperature is increased up to 80°C and then vaccume pump is started and permeation of furfural is carried out 267Pa.(2mm.hg.) as shown in table no. 2 After pervaporation we get a mixture of light fractions and furfural. For purification of furfural we used the process of simple distillation. This process was carried out for only 30 minute and 120ºC. Pure furfural was got as a residue and in distillate we get light fraction mixture. After whole process we confirmed concentration of furfural by using above volumetric estimation process. From which we get the maximum concentration of furfural as 99%. In above process we took the temperature of 120ºC because all light fractions were distilled and we get pure furfural.

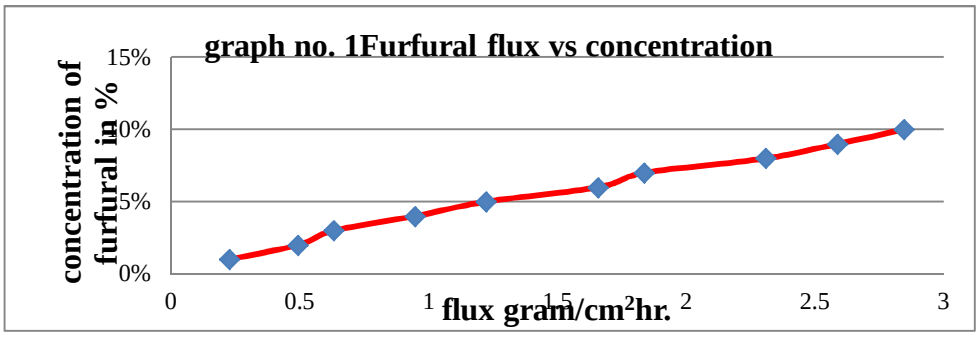
D. Pervaporation Process for Parameter Study

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Sr. no.	Weight of feed in grams	Feed concentration	Weight of permeate in grams	Flux of furfural in gram/cm^{2}hr.
1	200gram	1%	1.15	0.227
2	200gram	2%	2.5	0.494
3	200gram	3%	3.2	0.6324
4	200gram	4%	4.8	0.9486
5	200gram	5%	6.2	1.225
6	200gram	6%	8.4	1.66
7	200gram	7%	9.3	1.838
8	200gram	8%	11.7	2.31
9	200gram	9%	13.1	2.588
10	200gram	10%	14.4	2.8458

Observation table no.4 -of Pervaporation process for parameter study



For study of different parameter we were carried out the pervaporation process for different condition in above case we used same quantity of feed, constant temperature of pervaporation and constant time 1 Hr. for process. In above case we was taken different initial concentrations of furfural and fed to pervaporation set up. After pervaporation we get quantity of furfural in permeate and measured the flux rate of furfural permeation through PTFE membrane. From above observation we can say that if we increased the concentration of furfural in feed then the flux rate also increased. For denoting the effect we draw the graph between the feed concentration and flux rate of furfural permeation.

$$\text{flux} = \frac{\text{weight of permeate quantity in grams}}{(\text{time requred to permeatein Hr.}) \times (\text{cross section area of membrane})}$$

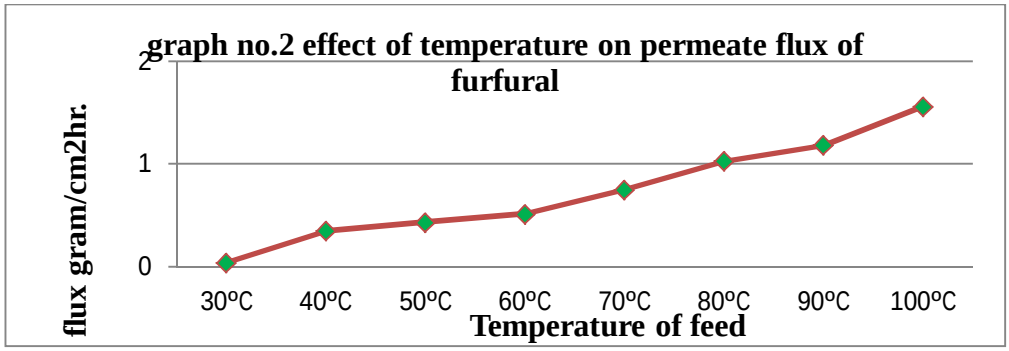
From above formula we can calculate flux of furfural permeation through PTFE membrane.. We were taken maximum concentration of furfural in feed 10% for that we get flux rate of 2.8458 gram/cm^{2}hr.

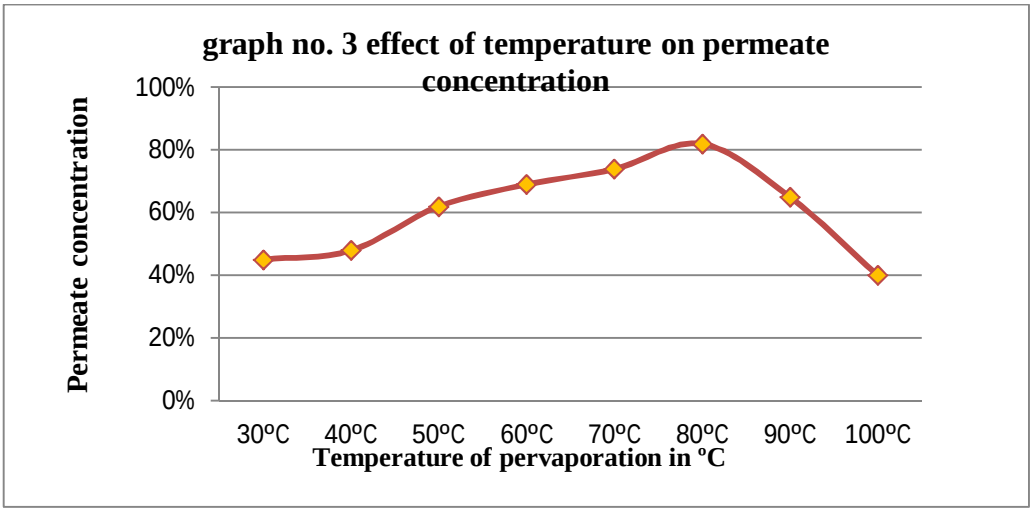
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Sr. no.	Feed taken for PV in grams	Temperature in ºC	Permeate quantity in grams	Permeate flux in $g/cm^2.h$	Concentration in permeate
1	200gram	30 ºC	0.2	0.039	45%
2	200gram	40 ºC	1.8	0.35	48%
3	200gram	50 ºC	2.2	0.434	62%
4	200gram	60 ºC	2.6	0.514	69%
5	200gram	70 ºC	3.8	0.751	74%
6	200gram	80 ºC	5.2	1.027	82%
7	200gram	90 ºC	6	1.185	65%
8	200gram	100 ºC	7.9	1.56	40%

Observation table no.5- of Pervaporation process for parameter study

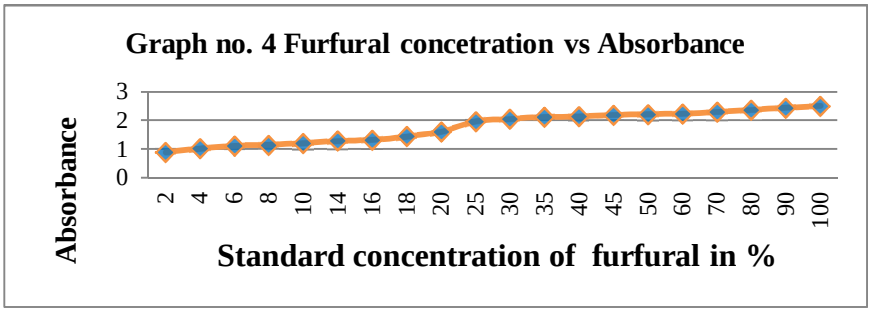




From above graph no. 2 characteristics we can say that if we increase the temperature of pervaporation then we get increased in permeate flux of furfural through PTFE membrane. The maximum flux rate gets at 100 ºC.From above graph no.3 characteristics we can say that if we increase the pervaporation temperature then the permeate concentration of furfural through PTFE membrane is also proportionally increased up to 80 ºC after that the permeate concentration was decreased because after 80 ºC the water Vapour will gets increases and then nature of membrane turns into hydrophilic and concentration of furfural gets decreased.

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E. Concentration for Conformation Determination Analysis Of Product



Here we were taken different standard concentrations of furfural as like as 2, 4, 6, 8,…....,up to 100% and measured the absorbance at constant 340nm wavelength for different concentrations. The maximum absorbance was found as 2.501 at 100% and minimum absorbance was found as 0.896 at 2%.

IV. CONCLUSIONS

Furfural was synthesized by acid hydrolysis process & its separation was done by using membrane separation techniques. Two methods of furfural estimation was studied which are by using spectrophotometer. In this work different parameters like effect of feed composition on permeation flux, effect of feed composition on furfural concentration in permeate , effect of temperature on permeate flux , effect of temperature on furfural concentration. Comparatively use of PV for the separation and high quality of furfural yield by using PTFE membrane also done. It can be thus concluded that if we increases the temperature of feed then the permeate flux through membrane is increased.

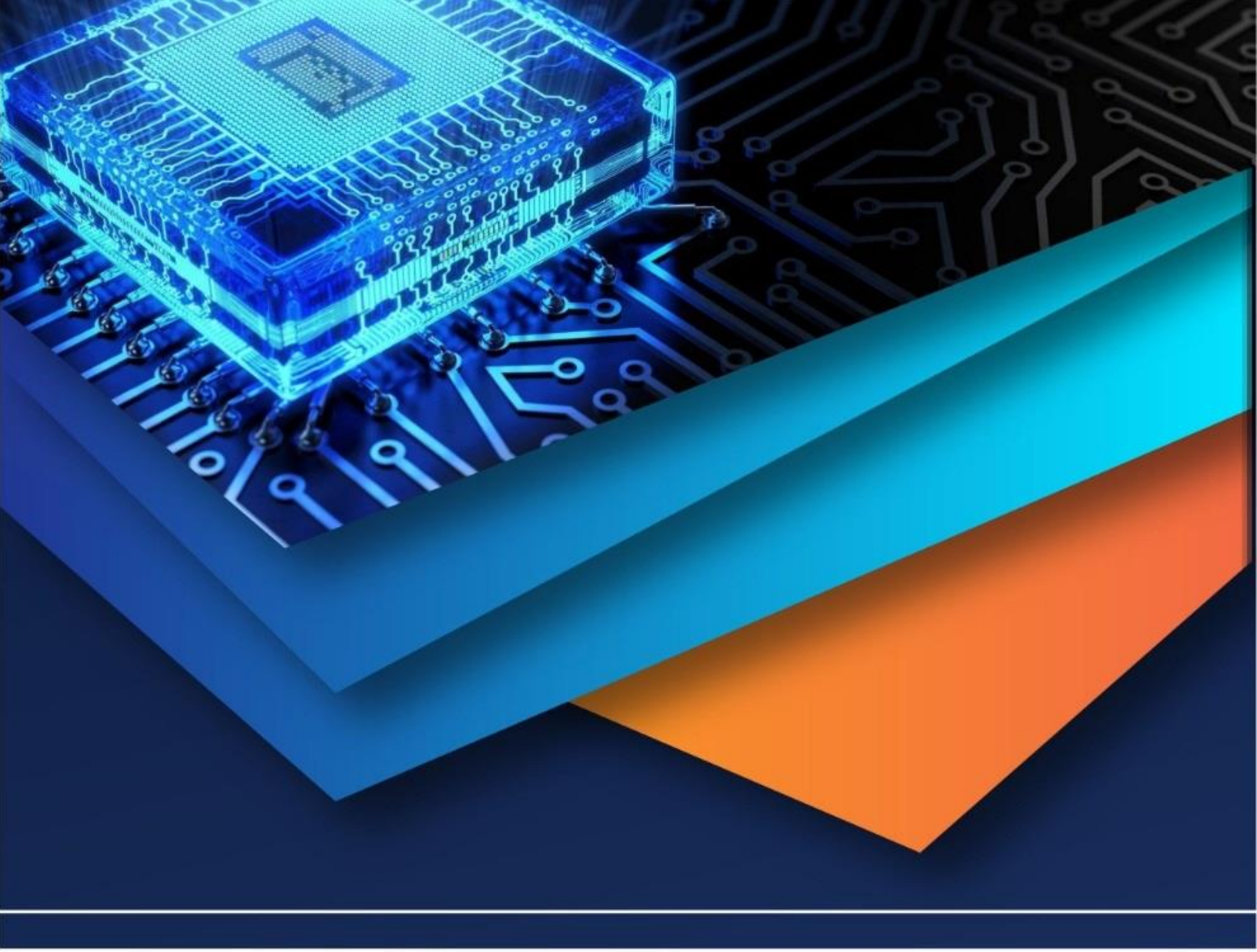
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