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Removal of Heavy Metal from Wastewater using Water Chestnut Shell as an Adsorbent

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Abstract: ***The contamination of water resources as a result of industrial activity is on the rise and is a global concern. The heavy metals found in wastewater are long lasting and non-biodegradable. Contamination with heavy metals over acceptable limits could result in major health problems. Chemical precipitation, chemical oxidation, ion exchange, membrane separation, reverse osmosis, electrodialysis, adsorption, and other technologies are used to lessen the influence of heavy metals on water bodies. Some procedures are extremely costly, energy-intensive, and frequently result in the production of harmful by-products. The use of adsorption as a cost-effective approach for removing heavy metals from industrial wastewater has been examined. The usage of Trapa bispinosa peels/shell as a low-cost adsorbent for wastewater treatment is discussed in this paper. Chemical activation was used to make activated carbons from Trapa bispinosa peels and shells. Activated carbons made from a combination of Trapa bispinosa peels/shells and Phosphoric acid with varying impregnation ratios. The results revealed that the activating temperature for the production of Trapa bispinosa-derived activated carbon is 500°C (AC). CHNS, X-ray diffraction (XRD), and Fourier transform infrared (FT-IR) spectroscopy were used to analyze the activated carbons.***
Keywords: Trapa bispinosa, water chestnuts, adsorption, wastewater, activated carbon.

I. INTRODUCTION

Environmental contamination is one of the most serious issues facing the globe today. It has been quickly expanding in recent years, reaching dangerous levels because to its negative impacts on living organisms. Toxic heavy metals are pollutants that have a direct impact on human and animal life. Environmental contamination is increasing day by day as a result of the enormous expansion in human population. Increased global industry and urbanization result in a continual release of harmful effluents into our environment. Heavy metals are one of the contaminants produced by several industrial processes that contain metal. These pollutants enter the biological cycle through drinking water, food, and the air we breathe (RENGE 2012). The entry of these contaminants into our biological cycle has the potential to harm human health. As a result, harmful heavy metal concentrations must be reduced to a tolerable level before being discharged into the environment. There are other ways for treating heavy metals; however, they are all more expensive than activated carbon adsorption. Thus, traces of heavy metals can be removed by adsorption on activated carbon made from abundant and inexpensive biomass material.

Heavy metals are a group of elements with metallic characteristics that aren't well defined. Transition metals, certain metalloids, lanthanides, and actinides are among the hazardous heavy metals. Heavy metals include copper, lead, and zinc, which are all common transition metals. These metals are discharged into the environment through a variety of causes, including leaded gasoline, industrial effluents, and acid rain leaching of metal ions from the soil into lakes and rivers (Singh 2011). The majority of heavy metals are produced by the electroplating industry. The petroleum refining industry, as well as the leather, tannery, textile, pigment & dyes, paint, and wood processing sectors, produce considerable amounts of heavy metals. Elements with an atomic density more than 6 g/cm^3 are classified as heavy metals (Akpor 2014).

A. Selenium

Selenium (Se) is an important micronutrient that is beneficial to our health at low concentrations, but it is harmful when consumed in excess of the daily recommended amount. Excess Se in the environment, on the other hand, creates substantial contamination in the soil, atmosphere, and water, as well as posing a serious risk to human health (Mineshi Sakamoto 2012). Selenium is essential for human health and should be consumed in daily doses of 40–400 ($\mu g\ d^{-1}$). Selenium deficiency can lead to cardiovascular, muscular, and liver disorders. GGI problems, hair loss, nail sloughing, weariness, irritability, and neurological impairment are also caused by concentrations more than 400 ($\mu g\ d^{-1}$) (Nabavi Larimi et al., 2016).

In India, se deficiency and toxicity have been documented. According to reports, selenium pollution is a problem in India's northwestern region.

Punjab is India's most dangerous state, with over 1000 hectares of poisoned agriculture. Se mobilization and easy bioavailability are caused by alkaline soil pH, growth of Se bio accumulators, and inappropriate treatment of industrial effluents/emissions. After China (28.5 %) and Thailand (34.5 %), India is the world's second-largest rice producer (21.7 %) and exporter (16.5%), with excessive Se (on average 157 ng/g) comparable to USA rice (180 ng/g).

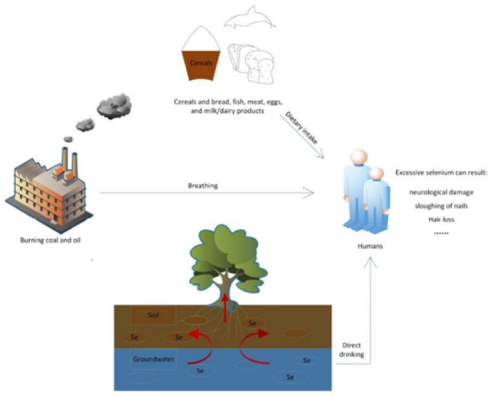

Fig 1. Schematic diagram shows the transfer of selenium from environment to human through daily life. (Yangzhuo Hea 2018)

B. Activated Carbon

The formation of micropores and mesopores, in particular, is critical because it permits porous carbons to adsorb vast amounts and a variety of chemical substances from gas or liquid streams. Physical activation and chemical activation are the two methods for making activated carbon. In physical activation, a raw material is first carbonized, then steam or carbon dioxide is used to activate the carbonized substance; there are two steps: carbonization and activation. Chemical activation involves impregnating a raw material with an activating reagent, then carbonizing the impregnated material at a high temperature in a muffle furnace (Sekirifa Mohamed Lamine a* 2014). For chemical activation, different activating agents such as potassium hydroxide (KOH), sodium hydroxide (NaOH), zinc chloride ($ZnCl_2$), potassium carbonate (K_2CO_3), and phosphoric acid (H_3PO_4) have been described. KOH causes only micro porosity to widen, resulting in more heterogeneous micropores, whereas $ZnCl_2$ causes wide micropores as well as low mesopores. H_3PO_4, on the other hand, develops not only large mesopores but also macropores.

II. MATERIALS AND METHODS

A. Materials Required for the Experimental Work

1) Raw material (water chestnut shells)

2) Chemicals: All chemicals were used of Analytical Grade (A.R), stock solution of Se(VI) (make: inorganic ventures, Germany) of 1018 ppm used for the preparation of synthetic working solutions. Desired concentrations were obtained by diluting stock solution.

B. Preparation of Material

Water caltrop shells are collected from a pond and washed thoroughly with deionized water to remove any contaminants. It is dried at 110^{o}C to remove the complete moisture from water caltrop.

Fig 2. Water chestnut shells before and after crushing and grinding

1) Impregnation process

Impregnation can be done by two processes

a) Physical process

b) Chemical process

We employed a chemical technique to expand the pore size in this experiment, and the raw material was phosphoric acid (H_3PO_4) with 88% purity.

We employed a 5:1 ratio to impregnate the water caltrop, which is a convenient ratio for the operation.

Fig 3. Water chestnut after impregnating with H_3PO_4

2) Activation Process

Activation is done two processes follows

a) Muffle furnace

b) Tubular furnace

It has been validated that muffle furnaces can provide proper activation at temperatures ranging from 400 to 700^oC; in this study, we used two temperatures of 400^oC and 500^oC depending on the features of activating agents such as H_3PO_4.

The activation of the water caltrop takes two hours, after which it can be left in the environment.

3) pH Neutralization Process: After activation, some impurities in the active carbon, such as phosphoric acid contamination, are shown to be more particularly phosphorous group, which collapses the morphological structure and entraps the porosity. The pH is neutralized to seven by ignoring the problem.

4) Drying Process: The pH neutralized water caltrop is dried at a temperature of 110^oC for 24 hours as the final phase of this process. The dry material is then crushed using a mortar pestle to produce fine activated carbon.

Fig 4. Water chestnut shell after activation

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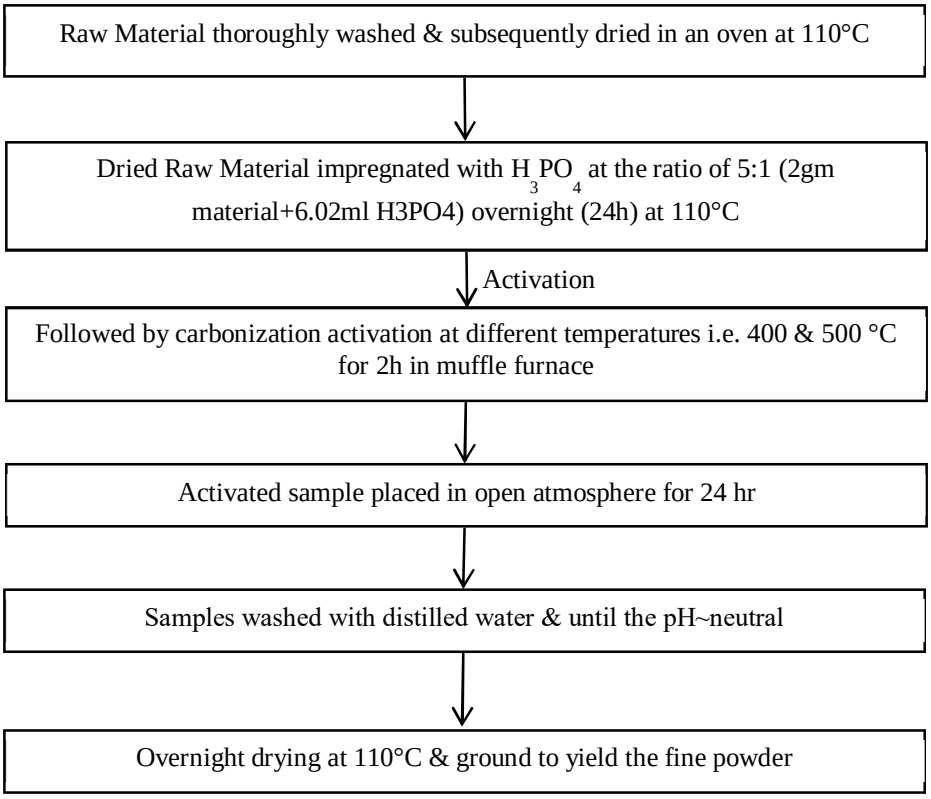
5) Flow chart for preparing activated carbon from water caltrop

For 400ºC and 500 ºC



III. CHARACTERIZATION

A. XRD (X-Ray Diffraction)

1) Preparation of sample

Determination of an unknown material, and samples

a) Grinding of sample into fine powder, usually in a fluid, to reduce producing excess strain (surface energy) that might skew peak positions and randomize orientation

b) A powder with a particle size of less than (188 mesh) is use

c) Place the sample in a sample holder or on the sample surface

d) To avoid interference, the material should be amorphous

e) If not producing an oriented spreading, care must be taken to establish a smooth upper surface and a random distribution of lattice orientation

f) For unit cell determinations, a small amount of a standard with known peak positions (that do not interfere with the sample) can be added and used to correct peak positions

B. FTIR (Fourier Transform Infrared Spectroscopy)

One can make a pellet out of powder through

1) The powder sample must be ground to a particle size of less than 5 mm in diameter for this procedure. Large particles, on the other hand, scatter the infrared beam and generate a sloping baseline in the spectrum.

2) Into an agate mortar, add a spatula full of ac and grind it to a fine powder until crystallites are no longer visible and it turns somewhat "pasty" and adheres to the mortar.

3) Take a little amount of powder sample (about 0.1-2%).

C. CH-NS Analysis

To supply carbonate and organic carbon, and to provide a sense of the composition WC, WCAC 400, WCAC 500 based on total organic carbon/total nitrogen [C/N] ratios, elemental analyses of total nitrogen, carbon, hydrogen, and Sulphur are done. A CHNS analyzer, model NA 1500 from Carlo Erba Instruments, is used to determine total nitrogen, carbon, and Sulphur. Freeze-dried and crushed samples are weighed (5-10 mg) and mixed with an oxidant (Vanadium Pentoxide [V_2O_5]) in a tin capsule, which is subsequently combusted in a reactor at 1000°C for the CHNS analysis. In a momentarily increased oxygen atmosphere, the sample and container melt, and the tin encourages a violent reaction (flash combustion). A steady flow of carrier gas (helium) transports the combustion products CO_2, SO_2, and NO_2 via a glass column containing a tungsten trioxide (WO_3) oxidation catalyst and a copper reducer, both maintained at 1000°C. Nitrogen oxide is converted to N_2 at this temperature. The N_2, CO_2, and SO_2 are then carried by helium to a 2-m-long packed column (Poropak Q/S 50/80 mesh) where they are separated and measured using a TCD (set at 290°C).

D. ICP-OES

The instrument as a stream of liquid sample. The liquid is turned to an aerosol inside the device by a process known as nebulization. The sample aerosol is then delivered to the plasma, which desolates, vaporizes, atomizes, and excites and/or ionizes it. Excited atoms and ions release their unique radiation, which is collected and sorted by wavelength by a device. The radiation is detected and translated into electronic signals, which the analyst can use to calculate concentrations.

IV. ADSORPTION EXPERIMENTS

A. ICP-OES

Activated Carbon screening study

1) Blank Initial Se concentration: 388.1 ppb
2) Blank Final Se concentration: 383.9 ppb
3) Temperature: 25.0°C
4) Reaction Time: 1h, 3h, and 6 h
5) Rpm: 150
6) Dose: 50mg/100ml

Table 1. Initial study of adsorbent on Se (VI)

Sr.No.	Sample Name	Sample Code	Conc. (ppb) after 1hr	Conc. (ppb) after 3hr	Conc.(ppb) after 6hr
1	AC_WC_H3PO4_400C	A	345.3	276.5	332.0
2	AC_WC_ H3PO4_500C	B	198.6	213.3	281.1

B. Activated Carbon dose

1) Blank Initial Se concentration: 374.5 ppb
2) Blank Final Se concentration: 385.2 ppb
3) Temperature: 25.0°C
4) Reaction Time: 2h,4h, and 6 h
5) Rpm: 150
6) Dose: mg/100ml

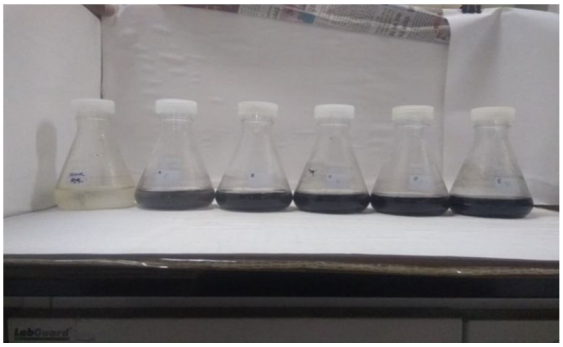


Fig 5. Dose Study Samples

Table 2. Dose Study of Adsorbent

Sr.No.	Doses Name (mg/100ml)	Sample Code	Conc. (ppb) after 2hr	Conc. (ppb) after 4hr	Conc.(ppb) after 6hr
1	25	A1	214.4	262.2	242.5
2	50	B1	217.7	259.3	232.5
3	75	C1	212.9	233.9	254.8
4	100	D1	229.2	277.9	253.9
5	125	E1	248.4	276.2	250.4
6	150	F1	257.4	285.2	266.0

V. RESULT AND DISCUSSION

A. *Yield of activated carbon*

Table 3. Nomenclature, preparation and yield of AC

Carbon name	H_3PO_4 (ratio)	Carbonization (temp ^oC)	Initial weight of precursor)	Final wt. of AC (gms)	Yield (%)
WCAC400^{o}C	5:1	400	2gm	0.809	40.45
WCAC500^{o}C	5:1	500	2gm	0.701	35.05

Table 3 summarizes the nomenclature preparation and yield of the AC impregnated with H_3PO_4 activation. The results clearly demonstrated that when the carbonization temperature rises, the material yield falls. The greatest visible percentage yield was percent, which was attained at a temperature of 400^{o}C.

B. *Activated Carbon Dose Analysis*

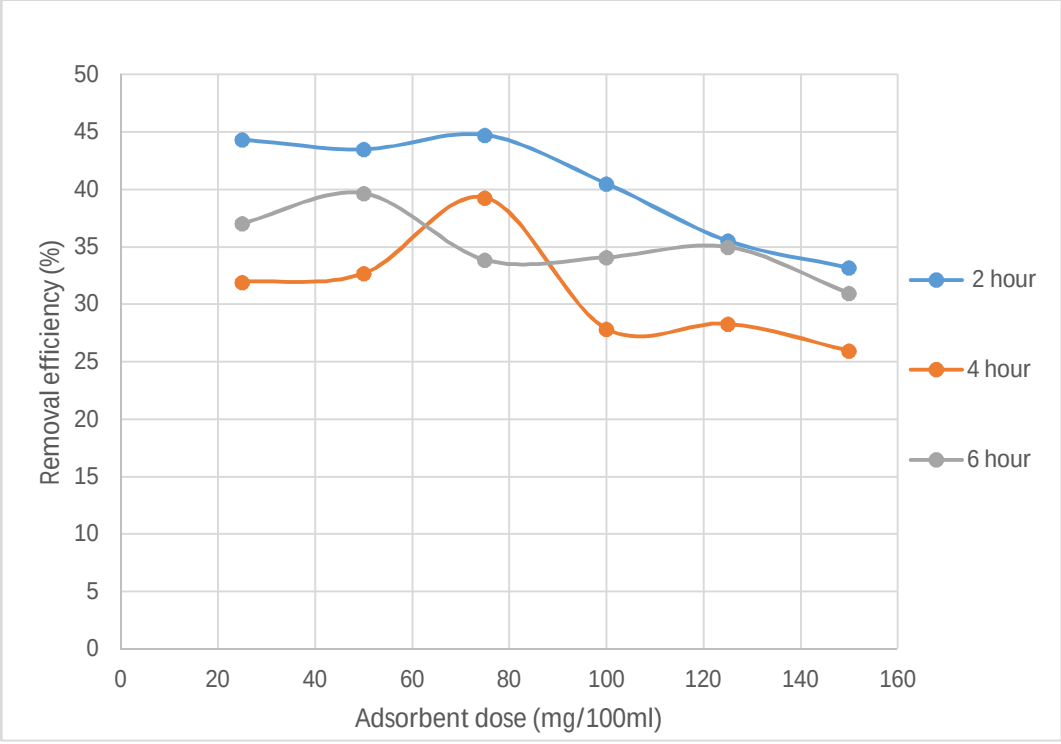


Fig 6. Efficiency vs Adsorbent dose proportion

C. Characterization of Activated Carbon

1) XRD: The figure shows X-ray diffraction for the WC AC MF 400°C and WC AC MF 500°C. The graph shows the diffraction peaks for WCAC 400°C at 24.2° and 43°, and 23° and 44° at 500°C. The XRD pattern reveals the typical amorphous form of activated carbon, with a carbon peak at 23°.

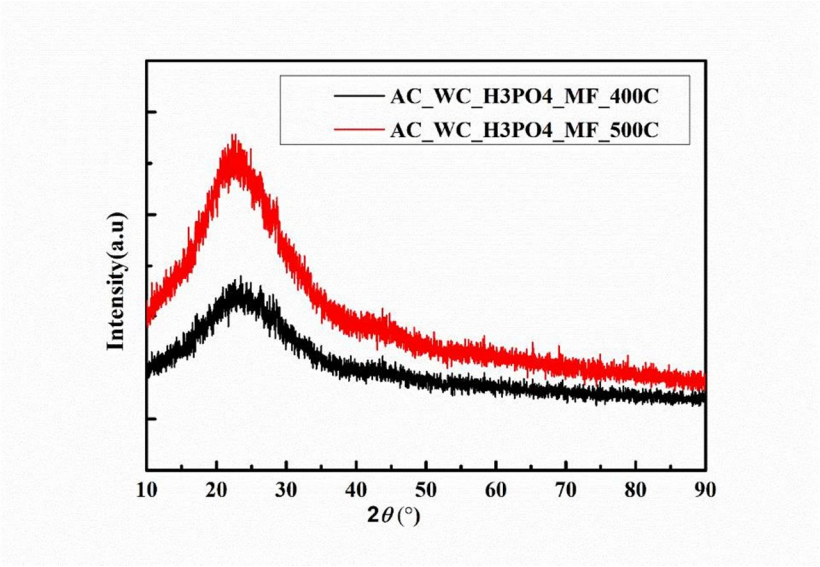


Fig 7. X-Ray diffraction proportion angle

2) FTIR: Figure shows an FTIR spectrum investigation of water chestnut shell powder WCAC 400 and WCAC 500. The hydrogen bond (–OH) group of alcohols and phenols has been assigned to the band in the range 3601.69 to 3855.45 cm^{-1} in WCAC 500. In WCAC, the 3635.47 cm^{-1} is usually a cellulosic OH-group.

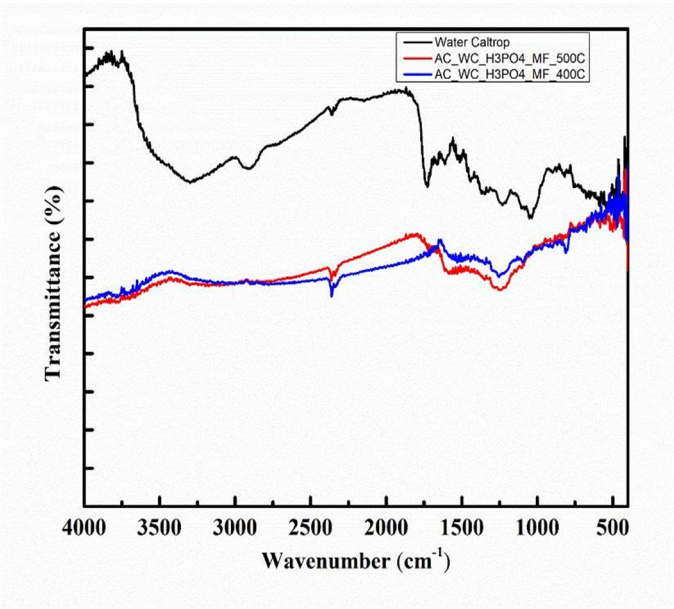


Fig 8. FTIR Spectrum

In WCAC 500°C, the strong absorption peak at 2358.49 cm^{-1} is indicative of the ethylene group. The presence of lactone was indicated by the other particular area identification in the blank WC, which were 1255.28. The hydrogen bond (–OH) group of alcohols and phenols has been attributed to the band in the range 3768.64 to 3916.33 cm^{-1} in WCAC 400°C. In WCAC, the 3635.47 cm^{-1} is usually a cellulosic OH-group. In WCAC 400°C, the strong absorption peak at 2342.42 cm^{-1} is indicative of the ethylene group.

3) CH-NS Analysis: The composition of the organic matter has been seen (i.e., based on total organic carbon/total nitrogen [C/N] ratios) water chestnut powder and prepared WCAC 400°C and WCAC 500°C was carried out in elemental analyzer. WC shells have high content of C (44.34%), H (5.763%), N (1.13%), C\N ratio 39.37 and C\H ratio 7.693. The activated carbon is impregnated with H_3PO_4 as an activating agent once it has been prepared. The carbon content was found to be 70.44% and 70.48% in WCAC 400 has nitrogen content 1.79% and hydrogen content was 2.330% with C\N ratio 39.2847 and C\H ratio 30.2285. For WCAC 500°C has 2.08% of N_2, 1.96% H, and 0.000% sulfur has C\H ratio of 35.9497, C\N ratio is 33.8571. It was found that after preparation of activated carbon the C\H ratio of WC is increased to 30.2285 for 400^{0}C and 35.9497 for 500^{0}C.

Table 4. CH-NS analyzed content

Elements	Water chestnut shell powder	WC_AC_MF_400^{0}C	WC_AC_MF_500^{0}C
Carbon (%)	44.34	70.44	70.48
Hydrogen (%)	5.763	2.330	1.961
Sulfur (%)	0.019	0.000	0.000
Nitrogen (%)	1.13	1.79	2.08
C\N ratio	39.3799	39.2847	33.8572
C\H ratio	7.6935	30.2285	35.9497

VI. CONCLUSION

Water chestnut shell was used to make activated carbon in this study. Water chestnut shell is a food processing sector industrial waste and an unique plant material with the ability to accumulate metals. This low-cost aquatic plant residue worked well as an adsorbent for removing Se (VI) from wastewater. Water chestnut is a popular floating herb in India and other parts of the world. H_3PO_4 impregnated the WC, which has a high porosity. When compared to water chestnut shell alone, WCAC has a higher acidic functional group and higher carbon content (70%). ICP-OES was used to investigate adsorbent qualities, which revealed a maximum removal efficiency of 44 to 45% for selenium removal from synthetic water with a very low adsorbent dosage for a 120-minute period. This water chestnut precursor may be used to make activated carbon, which is both inexpensive and abundantly available, making wastewater treatment possible. We conclude from the foregoing investigations that the water chestnut shell is a free biomass resource that can be used to make activated carbon, which can be used as an adsorbent to lessen the impact of industrial wastewater on aquatic systems and the environment. The efficiency can be enhanced through modification in future, which may develop the system to yield more than 50% that can give more stability and productivity.

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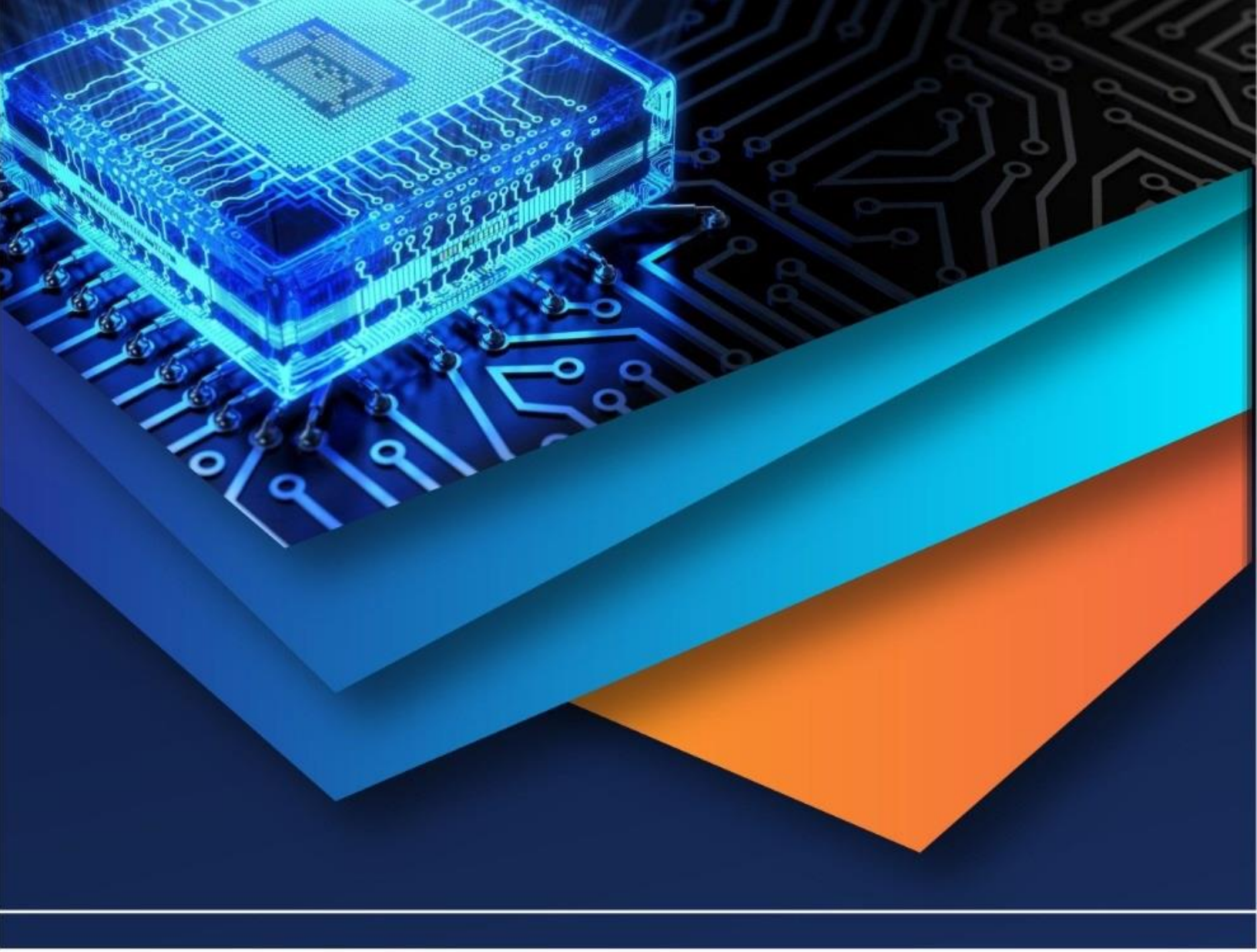

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