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Data Validation by FIS for Heavy Metal Ions Removal Using Husk of Cajanus Cajan in Aqueous Solution

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Abstract: Ever rising demands of electric and electronic appliances in the era of urbanization has increased the production of e-waste and menacing aqueous metallic wastes world-wide. The aqueous metallic waste contains high level of lethal ionic metals and compounds which need to be reduced to a level as suggested by various standards. In presented work; process of adsorptive removal of heavy metals mainly; Cr(VI), Cu(II) and As(III) by Husk of Cajanus Cajan (HCC) biosorbent; is used. To analyze the adsorptive efficiency of the HCC a batch mode experiment was conducted with the process parameters; pH of the solution (pH_0 = 1.0–7.0), Adsorbent Dose (AD = 0.1-0.7mg/50ml), Initial Metal Ion Concentration (IMC = 1–30 gm/L) and Temperature (Temp = 295-325 K). The results obtained through laboratory experimental setup were analyzed and it was observed that the removal of Cr(VI) is maximum in acidic pH range (pH_0=1), while the removal of Cu(II) and As(III) is maximum at neutral or near to neutral pH range (pH_0=6.0-7.0). The maximum removal of Cr(VI), Cu(II) and As(III) from the aqueous solution was done at high adsorbent dose (AD=0.7 gm/50 ml). The analysis also reveals that the pH of the solution is the most significant factor among all the experimental factors. To validate the obtained experimental results rules were induced using decision tree method and a Fuzzy Inference System (FIS) was designed and trained on the basis of rules thus obtained. Inferences given by the FIS, when applied on the same data, shows close accordance to the experimental results which validates the results and inferences obtained from laboratory experiments.

Keywords: Adsorpiton, FIS, Heavy metals, HCC.

I. INTRODUCTION

The race of urbanization will lead to the anticipated rise of various pollutants. Among various pollutants aqueous heavy metal contamination is the most menacing to the ecosystem. The heavy metals are non degradable and persistent in nature. Thus, the heavy metals are being accumulated in living organisms and water bodies [1-2]. The heavy metal concentration in human body may cause several ill effects, such as high concentration of Cu(II) may cause mental disorders, anemia, arthritis /rheumatoid arthritis, hypertension, nausea/vomiting, hyperactivity, schizophrenia, insomnia, autism, stuttering, postpartum psychosis, inflammation and enlargement of liver, heart problem, cystic fibrosis. High concentration of Cr(VI) causes skin rashes, respiratory problems, hemolysis, acute renal failure, weakened immune systems, kidney and liver damage, alteration of genetic material, lung cancer, pulmonary fibrosis. skin rashes, respiratory problems, hemolysis, acute renal failure, weakened immune systems, kidney and liver damage, alteration of genetic material, lung cancer, pulmonary fibrosis. Black foot disease, cardiovascular diseases, endocrine system disorders and cancer is caused by the lethal concentration of As(III) [3-4].

To reduce the risk of these fatal diseases it is required to regulate the concentration of metals into industrial discharge or wastewater. As per the CPCB the regulatory discharge limits in wastewater for these metals are 3.0 mg/L for Copper, 0.1 mg/L for Chromium and 0.2 mg/L for Arsenic[5]. The wastewater contains very high concentration of these heavy metals thus it is required to treat the wastewater before being dumped in to ground water. There are several methods for treatment but we find adsorption as one of the most cost-effective, energy effective sludge free and easy to operate method. Adsorption is efficaciously remove the heavy metal ions simultaneously under suitable condition [4].

Now days, well known charcoal or activated coal adsorbents are being substituted by various non-conventional adsorbents like biological waste material, nano materials, composites, polymers, clay and micro biomass [6-8]. Although charcoal very well known for their good adsorption efficiency and larger surface area,

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but its cost and limited availability made the search of other adsorbents liable [9]. In this study we are using the Husk of Cajanus cajan(HCC) as adsorbent of Cr(VI), Cu(II) and As(III) from their aqueous solution. HCC can be easily available in the local area because it is one of the popular Indian diets. We are using HCC as raw and unprocessed adsorbent to reduce the cost of process and make it as an economical but efficient wastewater treatment method.

To analyze the basic aspect of the adsorption process of Cr(VI), Cu(II) and As(III) from wastewater batch mode experiment set up was used. Process parameters considered in the presented study are pH of the solution(pH_0), Adsorbent dose(AD), Initial metal ion concentration(IMC) and Temperature(Temp) that affects the adsorption mechanism. Analysis carried out in this work helps to optimize these process parameters to get the optimum results from the experiment and helps to identify the full potential of HCC as adsorbent[10]. But the optimization of these process parameters does not provide adequate information to generate a full functioning adsorption wastewater treatment system. In today's scenario it is necessary to generate a system which is auto adjustable and fully functionalized. For generating such systems we need fuzzy logic approach that enables the system with human like thinking and decision making called artificial intelligence (AI). These methods use the input and output data set of the basic process as foundation of the system. AI based methods used mathematical techniques and logical thinking simultaneously. Thus they are capable of finding the relations between the various process parameters and predict the expected output for the set of applied process parameter within the limits. Hence it is evident that such methods are useful to reduce the time and cost of the research [11-12].

In the present study we developed a fuzzy inference system (FIS) for the empirical experiment and try to meet the today's requirement of an economical and effective wastewater treatment system.

II. MATERIALS AND METHODS

A. Preparation of Biosorbent

In this study the biosorbent was used without any activation, except sieving, to remove very fine particles. Husk of Cajanus Cajan (HCC) was collected from a local pulse mill of Harduaganj, Aligarh, U.P. HCC was washed, air dried and grinds to make a fine powder. It was sieved through 100µm-300µm; washed with double distilled water; Filtered and air dried. Lastly HCC was heated in an oven at 120 °C for 1 hr.

Proximate analysis and chemical analysis of the adsorbents were carried out as per ASTM. Bulk density was determined by using bulk density meter is the measurement of weight of adsorbent per volume (g/cm^3). Moisture content of the adsorbent was analyzed by using MB 50X moisture analyzer. Whereas the particle size analysis was carried out using standard sieves. FTIR spectrometer was employed to determine the presence of the functional group in HCC at room temperatures shown in Fig.1. Pellet technique was used for this purpose. HCC was also analyzed by Scanning Electron Microscopy (SEM) to show surface characteristics as shown in Fig.2 & Fig.3 before and after adsorption respectively. SEM is used to study the morphological features and surface characteristics such as surface texture and porosity of the HCC[13-14].

B. Preparation of Stock Solution

In the present work, the removal of heavy metal ions is done from their aqueous solutions. The aqueous solutions of Cr(VI), Cu(II) and As(III) are prepared synthetically by using various chemicals of analytical grade.

A stock solution of Cr(VI) was prepared by dissolving 0.2828g of potassium dichromate ($K_2Cr_2O_7$) in 1 L double distilled water. This solution contains hexavalent chromium concentration of 100mg/L. The desired concentration of Cr(VI) can be prepared from this stock solution, in agreement with Ahalya et.al. (2005). Stock solution of Cu(II) was prepared by dissolving 0.187g of copper nitrate anhydrous in 1 L distilled water, Li et.al. (2013). This solution contains the bivalent copper concentration of 100mg/L, and the desired concentration of copper can be prepared from this stock solution. For making As(III) stock solution, 1.7339g of sodium arsenite was dissolved in 1L distilled water, in accordance with Salim and Munekage (2009). This solution contains the As(III) concentration of 100mg/L. The pH of all these aqueous solutions is adjusted by the use of standard acid and base solutions [15-17].

C. Experimental Procedure

Adsorption studies were carried out in seven batch experiments and each batch has three set of Adjusting pH of the medium(pH_0) 1.0 -7.0, initial metal ion concentration(C_0) at 1.0 - 30 mg/L, adsorbent dose(D_0) at 0.1 - 0.7 mg/50 ml and at room temperature (T). The experiment was conducted by the synthetic aqueous solution in batch method. The batch sorption experiment was carried out in 250 ml iodine flask at room temperature with 50 ml of synthetic solution of metal ions at 150 rpm. After 120 min contact time iodine flask was removed and finally solution was allowed to settle down then filtered with whatman filter paper no.1. Filtrate was collected and taken in to account for metal ion estimation using atomic adsorption spectroscopy [16-17].

D. Fuzzy Inference System

Fuzzy logic is a multi-valued logic that signifies all possible values than bivalent logic or crisp set of data. This approach signifies the human like thinking and decision making. Fuzzy logic is applied for the systematic

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analysis and artificial intelligence application of any process[12]. This concept is accepting that things can be partly true and partly false at the same time. The imprecise perceptions effectively described by the linguistic variables and used to develop a fuzzy logic based computational system. This computational theory gives the human level machine intelligence. The developed computational theory is based upon the fuzzy rules calculations known as the Fuzzy Dependency and Command Language (FDCL).

FIS is a logical mapping generated between input data set and required output via a list of If- Then statements that are called rules. FIS interprets the input data, on the basis of defined rules and values to generate reasonable output. FIS decision-making depends upon the membership function on input variables. The membership function is truly satisfied if it varies between 0 and 1. But the beauty of fuzzy logic is that it has a variety of membership functions. These membership functions help to achieve human like perception based decision-making ability. The use of these membership functions on input variable data is called fuzzification of data. The FIS include following steps; Fuzzification of input variables, Apply the suitable fuzzy operator in the antecedent, Draw conclusion from the antecedent to the resultants, A collection of results across the rules, Defuzzification.

In this study we develop the FIS by defining the four input variables viz. pH of the solution (**pH_0**), Adsorbent dose (**AD**) gm/50 ml, Initial metal ion concentration (**IMC**) gm/L and Temperature(**Temp**) K. These variables are defined as per the empirical experiment data set as shown in Table I. The data set is divided in to three ranges: Low pH ranges from 1.0 to 3.0 pH, Medium pH ranges from 2.0 to 6.0 pH, and High pH range 5.0 to 7.0. In these values we find some overlapping values which are called Low and Medium or Medium and High at the same time. The FIS system for the present study was developed by the interaction of all the input variables to generate an output i.e. removal quantity of heavy metal ions from the aqueous solution. After defining the data set value now we fuzzified the data by Gaussian membership function as shown in equation 1. This function is defined by m and K, where K>0.

$$G_x = e^{-k(x-m)^2} \qquad .....(1)$$

The fuzzified data of all input variables are shown in Figure 1-4. Now after fuzzfication of data set we have to generate the decision tree by divide and conquer approach using persuasions related to the problem. This Decision-Tree further converts into commands using linguistic variables as If- Then statement sequences which are called as Fuzzy set rules. In this way we generated 81fuzzy rule set separately for Cr(VI), Cu(II) and As(III) removal from their

Table I Defined Input Variables for FIS

pH of the solution (pH)	Adsorbent Dose(AD) g/50ml	Initial Metal ion Concentration(IMC) g/L	Temperature (Temp) K
1.0	0.1	1	295
2.0	0.2	5	300
3.0	0.3	10	305
4.0	0.4	15	310
5.0	0.5	20	315
6.0	0.6	25	320
7.0	0.7	30	325

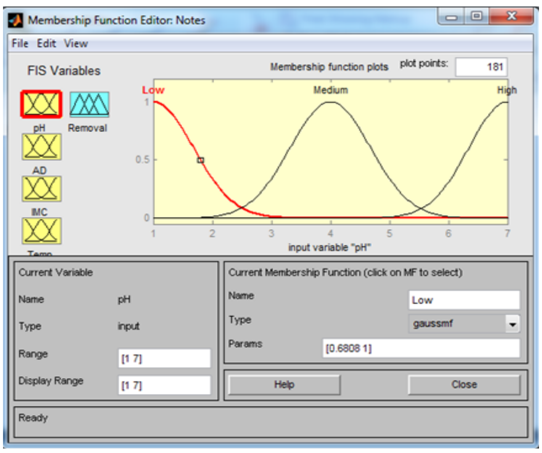

Fig. 1 Fuzzified Data of pH of the Solution

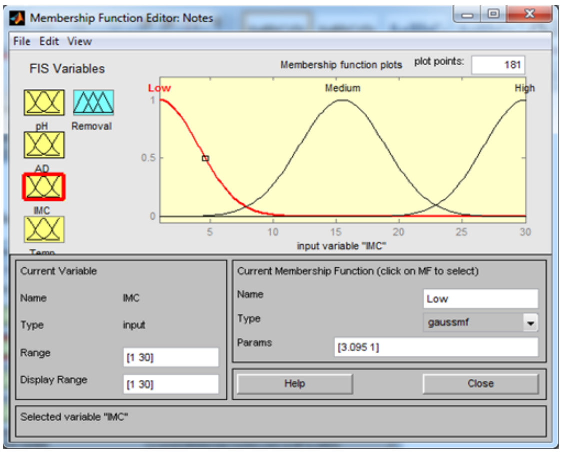

Fig. 2 Fuzzified Data of the AD

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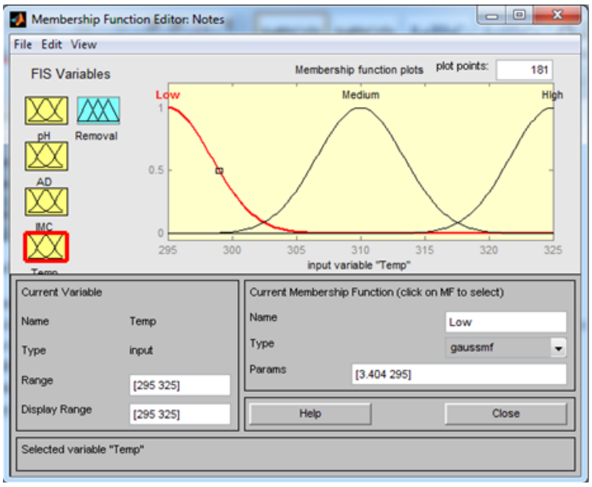

Fig. 3 Fuzzified Data of IMC

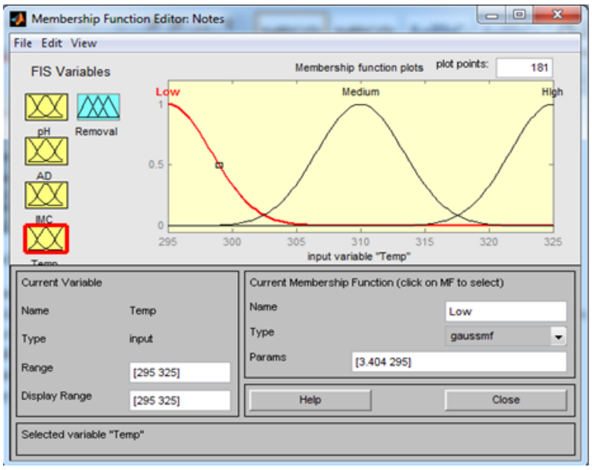

Fig. 4 Fuzzified Data of Temp

aqueous solution. These fuzzy rule set helps to develop the computable system of the present problem[18].Now the developed FIS was trained and evaluated for the required information.

III. RESULTS AND DISCUSSION

A. Characterization of Adsorbent

In this study we have analyzed HCC by various methods to analyze the suitability of HCC for adsorption. FTIR was used to identify the surface functional groups, SEM to check the surface morphology and iodine number to judge the adsorption capacity. The FTIR plots are shown in Fig. 5, that shows two curves one before adsorption and other for after adsorption. The analysis of this FTIR plots shows the peak at 3366.73 cm^{-1}due to presence of aromatic (-OH) and (-NH), and the peak at 2924 cm^{-1} due to stretching vibration of saturated alkane. The peak at 1623.74 cm^{-1} and 1445.8 cm^{-1} are due to stretching vibration of group (>C=C<). The peak at 1533.45 cm^{-1} is for (-NO) as (>CONHR) and peak at 1373.04 and 1032.15 is for medium stretching (>CN) vibration. The peaks between 860- 400 $cm^{-1}$are for aromatic (-CH) vibration. These functional groups act as suitable binding sites for the attachment of heavy metal ions. Functional groups on surface of HCC act as a proton donar or change its state with pH of the solution for the effective interaction between adsorbents and Cr(VI), Cu(II) and As(III). The presence of these functional groups is identified by the FTIR signals before adsorption. The FTIR images of HCC after adsorption for Cr(VI), Cu(II) and As (III) ions shows the shifting in signal peaks which is evidence of the successful adsorption of the Cr(VI), Cu(II) and As (III) ions onto the surface of adsorbents.

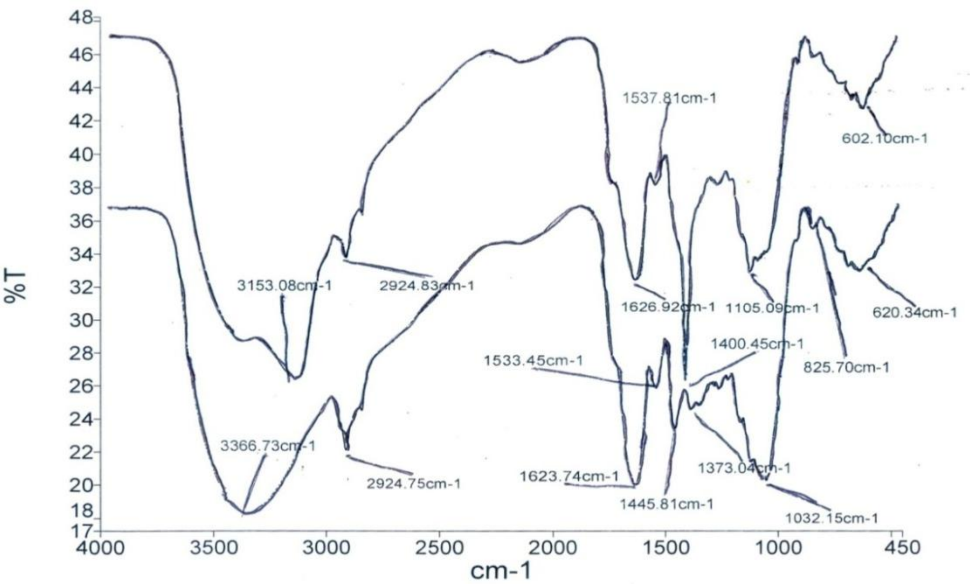

Fig. 5 FTIR Image of HCC Before and After Adsorption

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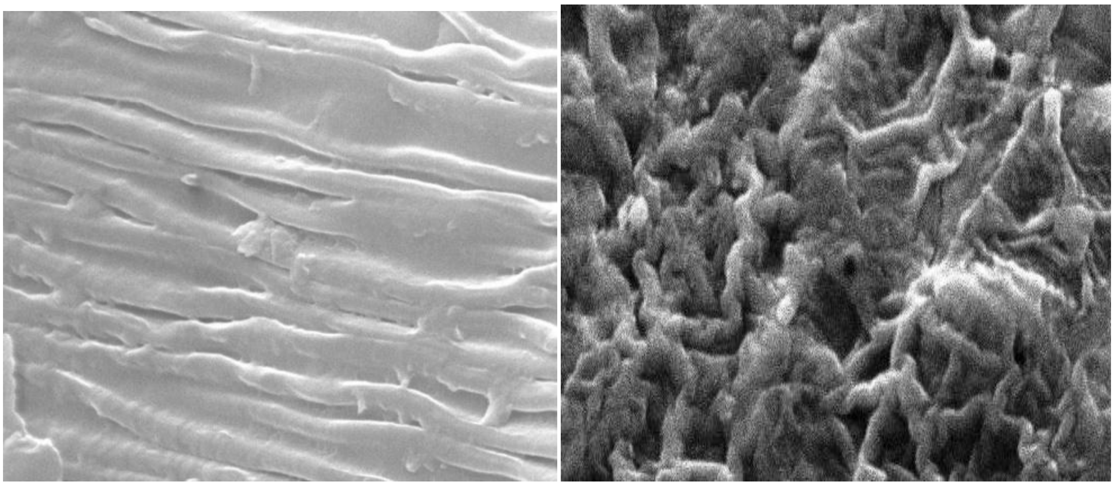
Scanning Electron Microscopy was also used to analyze the surface morphology of the HCC. This technique is useful to judge the agreement of the HCC surface texture and porosity for adsorption. The HCC was analyzed before and after adsorption to examine the change over the surface of the adsorbent. The Fig. 6(a) and Fig. 6(b) shows the SEM images of HCC before and after adsorption and these images reveals that the surface of HCC has grooves and pits, which makes this surface suitable for the adsorption of Cr(VI), Cu(II) and As(III) from aqueous solution.



(a) (b)

Fig 6. SEM Image of HCC (a)Before Adsorption (b) After Adsorption

The after adsorption SEM images of HCC show changes in surface morphology and conditions, which is evident from the fact that there was a successful attachment of Cr(VI), Cu(II) and As(III) onto the surface of HCC.

B. Results of Batch Optimization

This experiment was carried out in batch mode and use to optimize the all process parameters i.e. pH_0, AD, IMC and Temp. The defined range of pH was 1.0-7.0, the IMC was 1.0-30.0 gm/L, AD was 0.1- 0.7 gm/50ml and Temp was 295-325K. Each process parameter was optimized individually by keeping other parameters constant[19-21]. Batch optimization of Cr(VI), Cu(II) and As(III) was done separately by HCC in different batches and their results are as follows:

Optimization for Cr (VI), Cu(II) and As(III)

Adsorption of Cr(VI) by HCC was optimized for all process parameters pH_0, AD, IMC and Temp separately. The study shows that the pH_0 is varied from 1.0- 7.0 pH and the removal was found to be maximum at strong acidic medium i.e. 1.0-2.0 pH. At low pH value the concentration of protons are high in solution and other positively charged groups are present on adsorbent site. Thus, they easily attract the anions of Cr metal ions present in aqueous medium[10, 22].

For the removal of Cu(II) ions from its aqueous solution the pH value ranges from 1.0 – 7.0. The result of Cu(II) adsorption with respect to pH of the solution are shown in the Fig. 2. The results clearly show that the maximum removal of Cu(II) occurs between 5.0 to 6.0 pH . There is a decline in the adsorption of Cu(II) ions above 6.0 pH value[16,21]. This is because above 6.5 pH Cu(II)is precipitated as $Cu(OH)_2$. The optimization for As(III) was also done in similar manner. In aqueous solution the As(III) remains as neutral H_3AsO_3 and $H_2AsO_3^-$. Near 6.0-7.0 pH the adsorbent surface has negative charge so it easily attract the anion complex of As(III), and show maximum adsorption near neutral pH[23]. Thus we conclude that the value of $pH_0^{(Cr)}$ = 1.0-2.0pH, $pH_0^{(Cu)}$= 5.0-6.0 and $pH_0^{(As)}$ = 6.0-7.0 pH shown in Fig. 2.

To observe the impact of AD we have selected AD in the range 0.1gm/50ml- 0.7gm/50ml. the results reveals that the removal of Cr(VI) Cu(II) and As(III) metal ions increases, as the quantity of dose increase. This is because the adsorbent surface has adsorption sites to bind the metal ions, and as the number of binding sites increases with the increase in AD the removal of metal ions also increases. We have used MATLAB to generate the graphical view of the effect of process parameters. MATLAB provides us a advantage to analyze the interacted impact of two parameters on the result simultaneously. Fig. 7 shows the interaction of pH and AD on the percentage removal of the Cr(VI), Cu(II) and As(III)[13,15,17].

IMC is the third important factor IMC factor of Cr(VI) Cu(II) and As(III) adsorption and their concentration ranges from 1mg/L-30 mg/L. The study clearly indicates that the removal of metal ions increases with increasing concentration of metal ions up to a certain amount.

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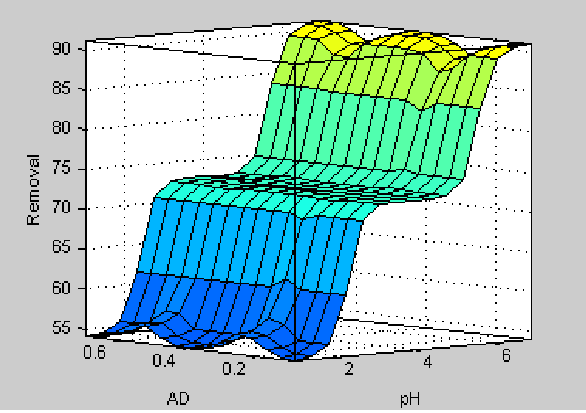
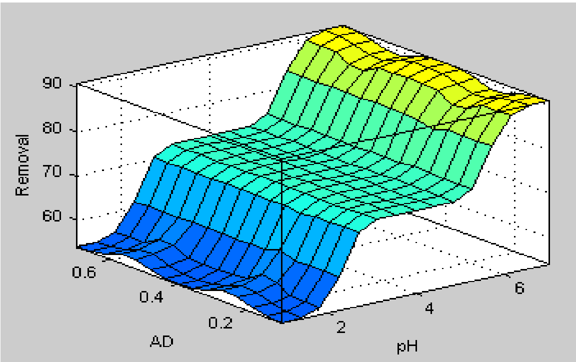
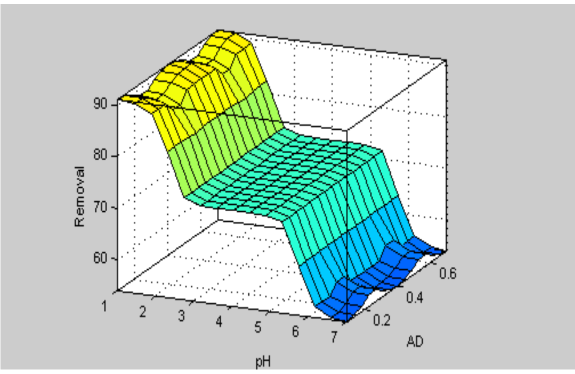


(a)



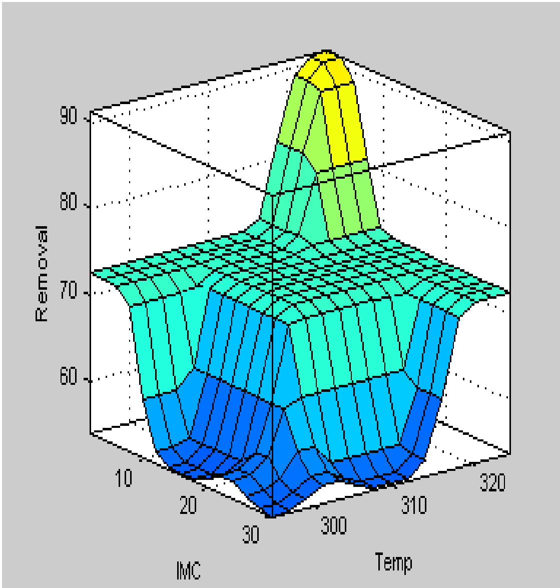
(b)



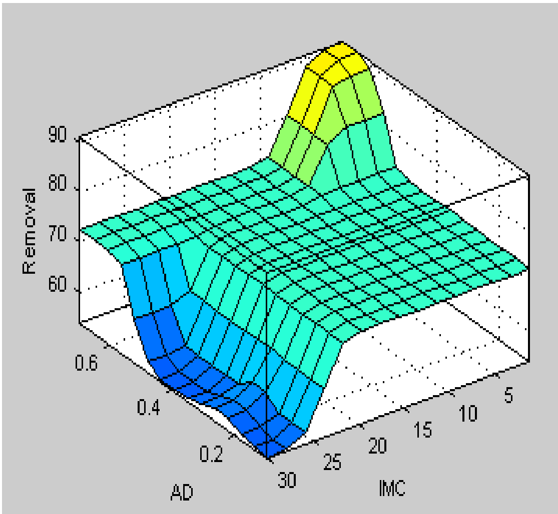
(c)

Fig. 7 Surface View of pH and AD for Cr(VI), Cu(II) and As(III) Removal.

Thereafter the further rise in concentration shows no significant effect on adsorption. The rise in removal percentage with increase in IMC is due to increase in the concentration gradient, which acts as a driving force for adsorption. This result has been shown in the Fig. 8(a), it shows the interaction of IMC with Temp factor for all the metal ions. This graph revels that the removal of metal ion is increased with the increase in temperature the removal is increased and it shows appreciable removal even at low concentration of metal ions. The maximum removal with respect to the available metal ion concentration is found at 320 K. Fig. 8(b) shows the interaction of IMC and AD process parameters and it reflects that the removal of meta ion get increased with the increase in the value of AD and it shows the highest removal with very low value of IMC. All these optimization results show full compatibility with the results of some related workers with some other adsorbents [22, 24].



(a)



(b)

Fig. 8 Surface View of the AD, IMC and Temp for the Removal of Cr(VI), Cu(II) and As(III).

C. Fuzzy Inference System Based Validation

The develop FIS contains 81 sets of rule base independently for Cr(VI), Cu(II) and As(III) and this rule base is the foundation stone of the successful FIS. The linguistic statement of the rule is as follows and it view as rule view in Fig. 9

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If **pH is Low, AD is Medium, IMC is Low, Temp is High** Then **Removal is High.**
If **pH is Low, AD is Medium, IMC is Low, Temp is Medium** Then **Removal is High.**
If **pH is Low, AD is Medium, IMC is Low, Temp is Low** Then **Removal is Medium.**
If **pH is Low, AD is Medium, IMC is Medium, Temp is High** Then **Removal is High.**
If **pH is Low, AD is Medium, IMC is Medium, Temp is Medium** Then **Removal is Medium.**

In this way we have developed the FIS for each metal ion and trained it successfully by generating the graphical view that represents in the Fig. 7 and Fig. 8. Now the FIS validation includes evaluation as the final step of process. It shows the final result of the generated fuzzy logic inference system. Evaluation of FIS follows the certain sequence of commands in the command prompt window of MATLAB. The evaluation of FIS shows the expected output by the set of input variable command. The expected output value shows the result without any experimental set up. It evaluates the expected output on the basis of designed FIS.

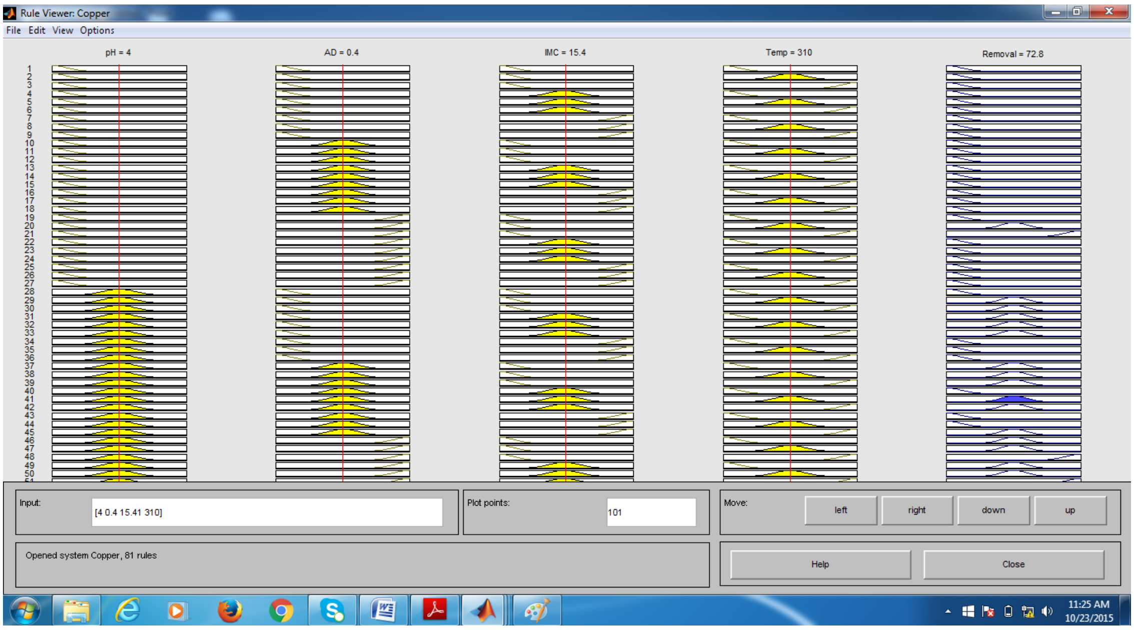

Fig. 9 Rule View of the If-Then Statement

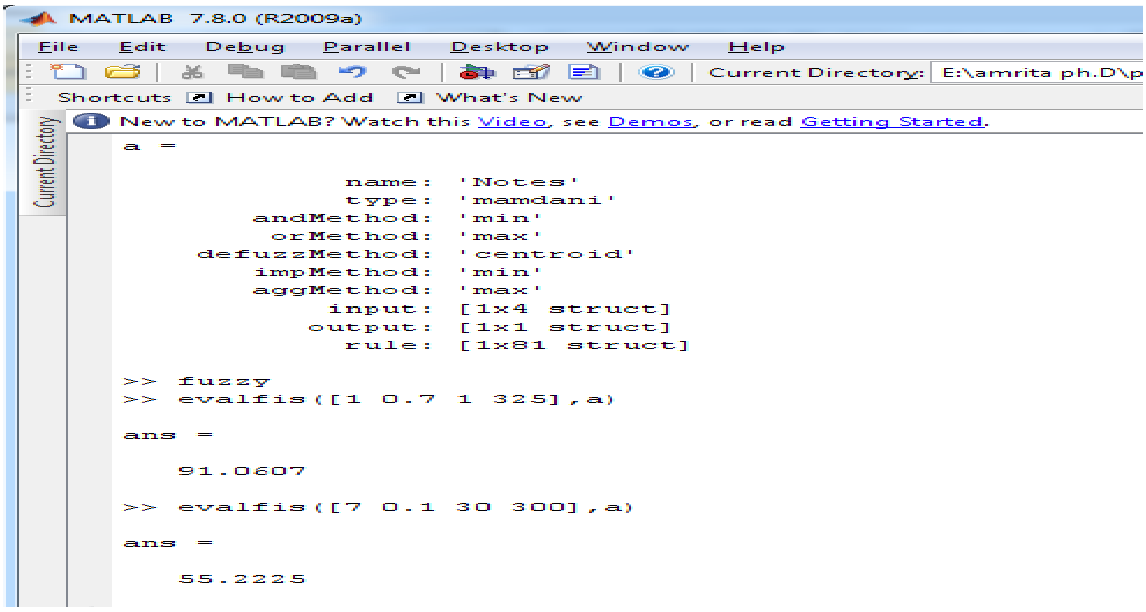

Fig. 10 FIS Evaluation Results in MATLAB.

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The evaluation of FIS of present study done by following certain set of command and evaluation command is as follows: >> a= readfis('Name of File)
>>evalfis [(pH, AD, IMC, Temp),a]

This command will generate the expected result of the input variable statement as removal quantity of the metal ion. We can put any value as input variables, but within the range of prescribed limits of FIS[25]. We can ask as many combinations as we can think and generate the expected output without any experimental setup as shown in Fig. 10. The result of FIS training shows the compatibility with the results of empirical study of the experiment. Training of FIS for Cr(VI) removal by HCC shows 91.0607 % adsorption at 1.0 pH value, 0.7gm/50ml, 1.0 gm/L and 325 K temperature. This result is in full agreement with the results of the empirical study. FIS evaluation of Cu(II) removal by HCC shows 91.1457% adsorption at 6.0 pH value, 0.7gm/50ml, 1.0 gm/L and 325 K temperature. Similarly the evaluation of As(III) removal by HCC also shows the full agreement with the given result of the experiment set up. The generated FIS is successfully evaluated and will be used as the reference of the further study without doing experimental analysis[18].

IV. CONCLUSION

The present study successfully optimized all the considered process parameters pH, AD, IMC and Temp for Cr(VI), Cu(II) and As(III) removal by HCC as adsorbent. The optimization results show the full agreement with the reference studies and the FIS of the study was successfully generated and trained for Cr(VI), Cu(II) and As(III) removal by HCC. The trained data of the FIS give expected results of empirical study of interacted mode of variable and individual variable by assuming other variables as constant. Thus, FIS is a useful tool to generate an intelligent wastewater treatment plant. The further study of this system will enable researchers to develop an approach of economical automated treatment plants using adsorptive removal of heavy metal ions with the non-conventional adsorbents.

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