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Comparative Analysis of Adsorption Efficiency Using Low Cost Adsorbents for COD Removal of Dye Intermediate (DASDA) Industrial Waste Water

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Abstract*— The manufacturing process of DASDA (4, 4'-Diaminostilbene- 2, 2'-disulfonic Acid) is conventionally carried out mainly by Sulfonation followed by Oxidation and Reduction. In this process iron powder and hydrochloric acid produces large quantity of solid ferrous chloride which is hazardous solid waste which creates a problem during the waste water treatment. After oxidation is carried out the filtrate contains 1.2- 1.5% DNSDA (4, 4'-dinitrostilbene 2, 2' – disulphonic acid) which goes along with the waste water stream to effluent treatment plant and increases COD level. The cost of treatment of effluent to decrease the COD adds up to cost of production. There is no replacement for this process so, one can either recover DNSDA or decrease the COD level which are generated due to the presence of DNSDA. Here experiments have been carried out and it has been shown that how cheap adsorbents such as activated carbon and lignite powder can decrease the COD levels. With a change in conventional effluent treatment flow sheet the COD is reduced substantially at the first stage facilitating further treatment. These results are compared to show effect of time of contact, quality of adsorbent and nature of effluent which affects the COD level. We are trying to approach a cleaner production method which is more suitable method in order to get a clean manufacturing process with reduced waste.

Index Terms— **Adsorption Efficiency, Cleaner Production Approach, DASDA, Low Cost Adsorbents.**

I. INTRODUCTION

The term Cleaner Production was defined by UNEP (United Nations Environment Program) in 1990 as: *"The continuous application of an integrated environmental strategy to processes, products and services to increase efficiency and reduce risks to humans and the environment"*. This definition has been used as the working definition of all programmes related to the promotion of cleaner production and still continues to be a valid definition.

A. Extracting the key elements from the definition of cleaner production, the following points come up:-

- Cleaner production entails a continuous process; it is not a one-time activity.
- Cleaner production moves towards striking a balance between the availability and consumption of materials (including water) and energy. It does not deny growth, but does insist that it be ecologically sustainable.
- Cleaner production refers to the approach of producing goods and providing services with a minimum of environmental impacts, given the technological and economic limits at the current time. It is not merely limited to minimization of wastes; rather it employs a broader context, and uses the term "impacts" in the life cycle.
- In addition to life cycle impacts, cleaner production also addresses health and safety concerns and emphasizes risk reduction. In this perspective, cleaner production is a holistic environmental strategy.
- Cleaner production is both efficient (in terms of increased outputs on an immediate basis) and effective (in terms of positive outcomes over the long-term).

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II. DASDA MANUFACTURING PROCESS

1. SULPHONATION:-Take 1000 kgs PNT in close lead reactor while stirring continuously. Now slowly add 2500 to 2700 kgs oleum (23 %) by continuously maintaining the temperature 95 ^{0}c to 100 ^{0}c. When oleum additions is over then check mass with PNT test, if it is ok start cooling up to room temperature then filter it out. The product is called PNTOSA and mother liquor is spent acid.
2. DUMPING AND FILTRATION: - The sulfomass formed is dumped at controlled rate. The whole sulfomass is dumped in 2 lots. Each lot takes 12 – 14 hrs and temperature is maintained 50^{0}c. After dumping is over, mass is pumped to nutsche filter. The filtrate known as spent acid is collected in a tank. The same is used in isolation process and remaining is commercially sellable. The filtration operation takes about 3 to 4 hrs. Cake obtained is PNTOSA, which is then centrifuged and sent for further process in oxidation vessel.
3. CHLORINATION:-Take 1200 kgs (100%) NaOH in hypo manufacturing tank and add 900 kgs ice and slowly charge 600 kgs chlorine. During this reaction temperature is maintained below 10^{0}c by ice to make pH 7 to 7.5.
4. OXIDATION:-Take about 10000 lit of tap water in brick lined vessel, heat upto 90^{0}c and add previously obtained PNTOSA. Make solution neutral by adding 600 kgs of soda ash. Now slowly add prepared hypo chloride solution. At this time add 300 to 400 kgs NaOH (100%). When hypo charging is over then add 10% salt of total volume, cool it down to room temperature and filter out by nutsche. Collect mass as DNSDA and mother liquor run out in ETP.
5. REDUCTION:-Take approximately 10000 lit tap water in reduction tank and heat it up to 95^{0}c temperature. Now add 650 to 700 kgs iron powder (Fe) and add HCl to adjust pH to 2. Start stirring and addition of above collected DNSDA. When reductions are completely over then add 40 to650 kgs of NaOH (100%). Filter out to clarify. Take, filtrate and transfer it to isolation tank.
6. SEPARATION OF DASDA: - The whole slurry from the reduction vessel is pumped to a plate and frame filter press. The filtrate is DASDA, which is taken to isolation vessel. The iron sludge is further washed with steam to remove maximum amount of DASDA.
7. ISOLATION:-Take above reduction filter mass and slowly charge spent acid to CR+ve (congo red test). There should be continuous stirring. By circulating steam the temperature of the reaction mass is brought up to 70^{0}c. Then filter out the mass by filter press. This product is DASDA.
8. FILTRATION:-After isolation the whole mass is then passed through the filter press. The cake from filter press is then sent to centrifuge and filtrate liquor goes to ETP.
9. CENTRIFUGE AND DRYING:-After the filtration is over the cake is centrifuged and then centrifuged mass is filled in the tray for drying. The dried mass is then sent to pulveriser. Product as per requirement in cake form from centrifuge or powder form after drying is sent for packaging.

A. *Process Flow Diagram:-*

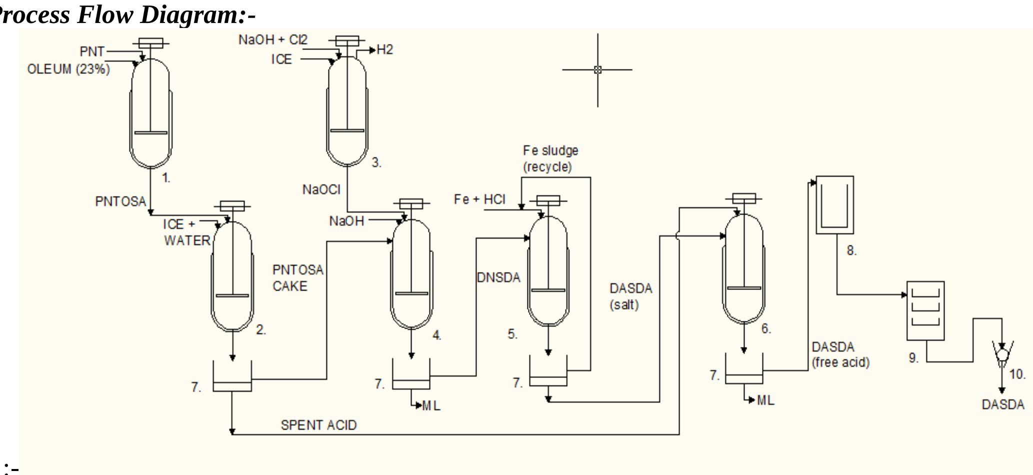

Fig 1:-

Sr. No.	Unit operation/Process	Sr. No.	Unit operation/Process
1.	Sulphonation	6.	Isolation
2.	Dumping	7.	Nutche filter/filter press
3.	Chlorination	8.	Centrifuge

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4.	Oxidation	9.	Tray Dryer
5.	Reduction	10.	Pulveriser

B. ETP:-

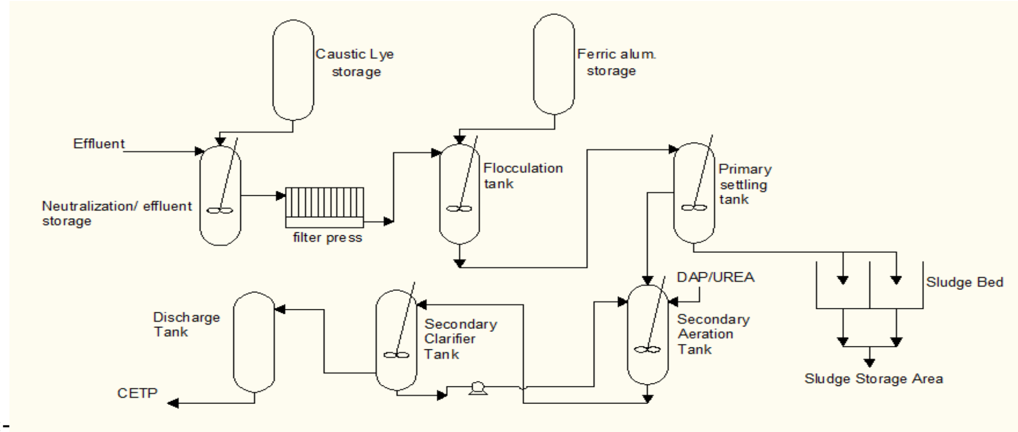

Fig 2:-

The Effluent Treatment Plant (ETP) is shown in above figure. First the effluents from all stages are collected in a storage/neutralization tank where caustic lye is added for neutralization. After neutralization the stream is passed through filter press to remove solids and passed to the flocculation tank. In the flocculation tank Ferric alum is added as a flocculating agent. After flocculation the mass is transferred to primary settling tank where sludge is separated to sludge-bed which is then sent to sludge storage area. The liquid from primary settling tank is sent to secondary aeration tank where DAP/Urea is added to decrease the COD level. The mass is transferred to the secondary clarifier tank for further aeration to decrease the COD level. After secondary clarifier tank if the COD level does not decrease then the mass is transferred again to the aeration tank where it is treated again with DAP/Urea. Once the COD level goes bellow GPCB norms i.e., below 3000, it is stored in discharge tank and sent to CETP for further treatment. The inlet stream of ETP has COD level ranging from 9000 – 10000 mg/lit, while after treatment in the ETP COD level comes to range of 6000 – 7000 mg/lit. So, to bring COD level below 3000 mg/lit re-treatment is done before sending to CETP.

III.METHODOLOGY

The conventional flow sheet of waste water treatment used by mist of the industrial units includes primary treatment, followed by secondary and tertiary treatments. During primary treatment, neutralization of the wastewater results in to increase of salts. Salts in high concentration inhibit biological activity and may cause an increase in non-settleable suspended solids in the treated wastewater. The flow sheet given subsequently is therefore proposed, wherein adsorption with inexpensive adsorbents could be employed, prior to the conventional primary treatment for increasing efficiency of the biological treatment.

Wastewater →	pH adjustment	→	Physicochemical/ Biological treatment	→	→ Adsorption		Treated Wastewater

This is expected to reduce refractory organics COD as well as BOD of the wastewater substantially at the first stage of wastewater treatment itself facilitating further treatment. From multi-stage production processes used in the production of organic chemicals, wastewater stream is likely to contain several organics as well as inorganic substances including products, intermediates, side products, by products and un-reacted raw materials.

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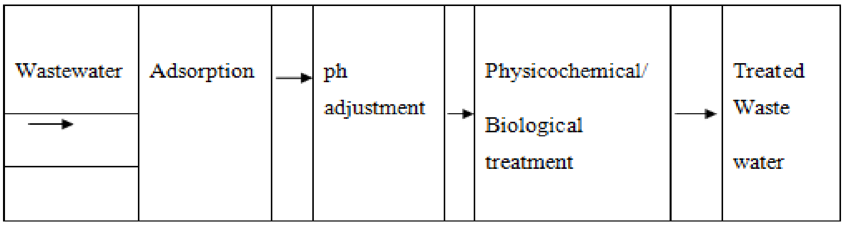

The industrial waste water being highly complex and varying widely in the absorbability of the compounds present the detailed analysis of such wastewater streams and study of their adsorption on different adsorbents was very complex. It was therefore decided to consider COD as the measured. These parameters COD and colour reflect the practical aspects of wastewater treatment on the industrial scale. Thus such a study should be of much relevance to the industry in selecting cost effective wastewater treatment technique for complying with the statutory regulations. The approach followed in the evaluation of adsorption performance of different adsorbents viz. Activated carbon, lignite in the treatment of waste water is thus based on detection of COD and colour in the wastewater with varying degree of treatment. This approach is entirely different from that followed by many of the researches who prepare wastewater samples from known solutes in the laboratory and carry out chemical analysis of the treated sample.

A. EXPERIMENTAL PROCEDURE

For the contact time experiments, waste water sample from Dyes intermediate plant were collected in labelled carboys and the carboys were then sealed. The samples are of ETP inlet stream and ETP outlet / CETP inlet stream. These samples were directly from the process plant streams before these had any chance of getting mixed with any other stream. In most of the cases these were concentrated streams, often referred as mother liquor. While carrying out experimental studies on the waste water from each carboy sample was analyzed for pH and COD. During experiment 200ml of sample is to be taken from the respective carboy in a cylindrical flask. In which 1% (2gm) Activated carbon (A/C) is to be added in to the flask and magnetic stirrer is started. 5-10 ml of sample is to be drawn in time interval of 10, 20, 30, 45, 60, 90, 120 min from this mass. Then Filtered on filter paper and the filtrate analyzed for COD. At the end of 2hr the stirring is stopped and the experiment is terminated. The experiments are to be repeated with 2% A/C, 3% A/C and 4% A/C, and similar procedure follow for Lignite adsorbent. All the experiments are to be carried out at room temperature of around 30^{o}C.

B. Experimental results

Experimental observations for Adsorption of Acidic ETP inlet sample.

Table 1:- Sample (200ml) with 1 % activated carbon (2 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	% COD reduction
0	10.5	7.8	0.255	8000	10	550.8	11016	0
10	10.5	8	0.255	8000	10	510	10200	7.40
20	10.5	8.2	0.255	8000	10	469.2	9384	14.81
30	10.5	8.3	0.255	8000	10	448.8	8976	18.51
40	10.5	8.4	0.255	8000	10	428.4	8568	22.22
50	10.5	8.5	0.255	8000	10	408	8160	25.92
60	10.5	8.5	0.255	8000	10	408	8160	25.92
90	10.5	8.8	0.255	8000	10	346.8	6936	37.03
120	10.5	8.9	0.255	8000	10	326.4	6528	40.74

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Table 2:- Sample (200ml) with 2 % activated carbon (4 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	% COD reduction
0	10.4	7.8	0.255	8000	10	530.4	10608	0
10	10.4	8.1	0.255	8000	10	469.2	9384	11.53
20	10.4	8.2	0.255	8000	10	448.8	8976	15.38
30	10.4	8.4	0.255	8000	10	408	8160	23.07
40	10.4	8.5	0.255	8000	10	387.6	7752	26.92
50	10.4	8.6	0.255	8000	10	367.2	7344	30.76
60	10.4	8.7	0.255	8000	10	346.8	6936	34.61
90	10.4	9	0.255	8000	10	285.6	5712	46.15
120	10.4	9.2	0.255	8000	10	244.8	4896	53.84

Table 3:- Sample (200ml) with 3 % activated carbon (6 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	% COD reduction
0	10.6	8.1	0.255	8000	10	510	10200	0
10	10.6	8.4	0.255	8000	10	448.8	8976	12
20	10.6	8.6	0.255	8000	10	408	8160	20
30	10.6	8.7	0.255	8000	10	387.6	7752	24
40	10.6	8.9	0.255	8000	10	346.8	6936	32
50	10.6	9	0.255	8000	10	326.4	6528	36
60	10.6	9.1	0.255	8000	10	306	6120	40
90	10.6	9.4	0.255	8000	10	244.8	4896	52
120	10.6	9.6	0.255	8000	10	204	4080	60

Table 4:- Sample (200ml) with 4 % activated carbon (8 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	% COD reduction
0	10.6	8.1	0.255	8000	10	510	10200	0
10	10.6	8.6	0.255	8000	10	408	8160	20
20	10.6	8.8	0.255	8000	10	367.2	7344	28
30	10.6	9	0.255	8000	10	326.4	6528	36
40	10.6	9.1	0.255	8000	10	306	6120	40
50	10.6	9.3	0.255	8000	10	265.2	5304	48
60	10.6	9.4	0.255	8000	10	244.8	4896	52
90	10.6	9.7	0.255	8000	10	183.6	3672	64
120	10.6	9.9	0.255	8000	10	142.8	2856	72

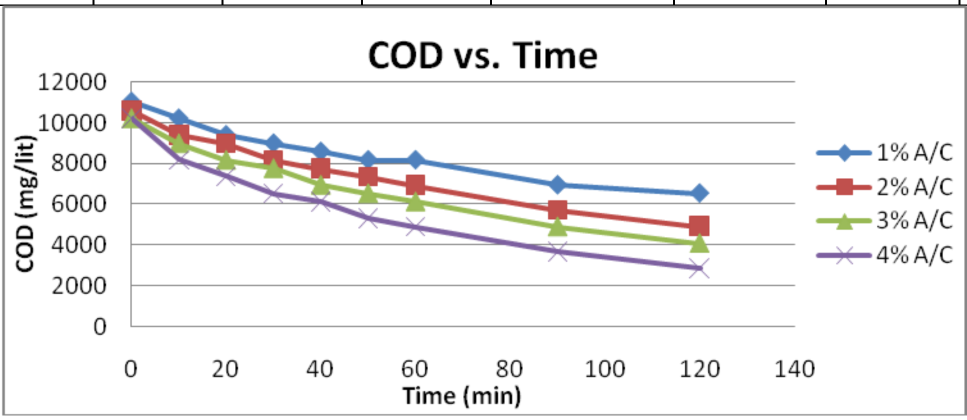

Fig 3

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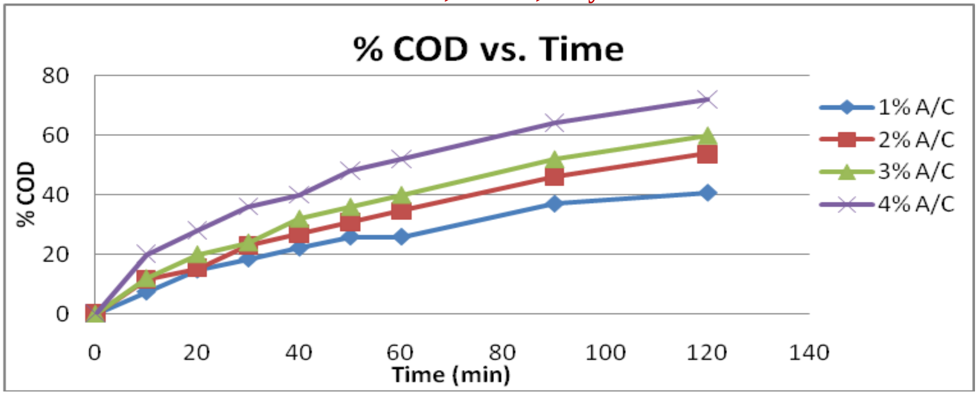

Fig 4

Table 5:- Sample (200ml) with 1% Lignite powder (2 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	% COD reduction
0	11.2	8.7	0.255	8000	10	510	10200	0
10	11.2	8.9	0.255	8000	10	469.2	9384	8
20	11.2	9.2	0.255	8000	10	408	8160	20
30	11.2	9.4	0.255	8000	10	367.2	7344	28
40	11.2	9.5	0.255	8000	10	346.8	6936	32
50	11.2	9.8	0.255	8000	10	285.6	5712	44
60	11.2	9.9	0.255	8000	10	265.2	5304	48
90	11.2	10.1	0.255	8000	10	224.4	4488	56
120	11.2	10.3	0.255	8000	10	183.6	3672	64

Table 6:- Sample (200ml) with 2% Lignite powder (4 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	% COD reduction
0	11	8.5	0.255	8000	10	510	10200	0
10	11	8.8	0.255	8000	10	448.8	8976	12
20	11	9.1	0.255	8000	10	387.6	7752	24
30	11	9.3	0.255	8000	10	346.8	6936	32
40	11	9.5	0.255	8000	10	306	6120	40
50	11	9.8	0.255	8000	10	244.8	4896	52
60	11	9.9	0.255	8000	10	224.4	4488	56
90	11	10.1	0.255	8000	10	183.6	3672	64
120	11	10.3	0.255	8000	10	142.8	2856	72

Table 7:- Sample (200ml) with 3% Lignite powder (6 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	% COD reduction
0	10.7	8.22	0.255	8000	10	505.92	10118.4	0
10	10.7	8.7	0.255	8000	10	408	8160	19.35484
20	10.7	8.9	0.255	8000	10	367.2	7344	27.41935
30	10.7	9.1	0.255	8000	10	326.4	6528	35.48387
40	10.7	9.4	0.255	8000	10	265.2	5304	47.58065
50	10.7	9.7	0.255	8000	10	204	4080	59.67742
60	10.7	9.84	0.255	8000	10	175.44	3508.8	65.32258
90	10.7	10.1	0.255	8000	10	122.4	2448	75.80645
120	10.7	10.3	0.255	8000	10	81.6	1632	83.87097

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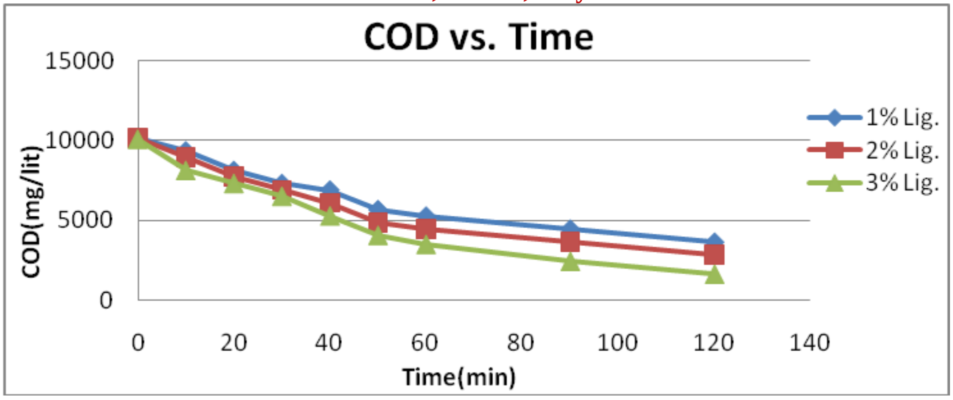

Fig 5

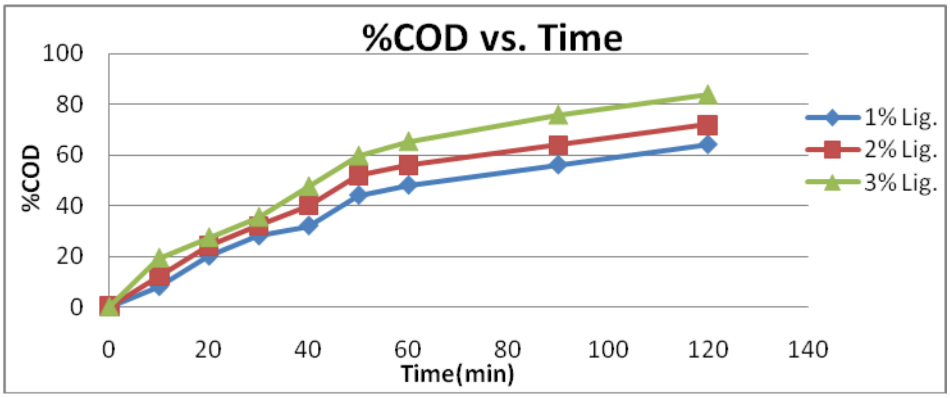

Fig 6

Experimental observations for Adsorption of neutral ETP inlet sample

Table 8:- Sample (200ml) with 1 % activated carbon (2 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	%COD reduction
0	10.6	8.44	0.255	8000	10	440.64	8812.8	0
10	10.6	8.65	0.255	8000	10	397.8	7956	9.722222222
20	10.6	8.83	0.255	8000	10	361.08	7221.6	18.05555556
30	10.6	9.02	0.255	8000	10	322.32	6446.4	26.85185185
40	10.6	9.16	0.255	8000	10	293.76	5875.2	33.33333333
50	10.6	9.3	0.255	8000	10	265.2	5304	39.81481481
60	10.6	9.4	0.255	8000	10	244.8	4896	44.44444444
90	10.6	9.6	0.255	8000	10	204	4080	53.7037037
120	10.6	9.7	0.255	8000	10	183.6	3672	58.33333333

Table 9:- Sample (200ml) with 2 % activated carbon (4 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	%COD reduction
0	10.6	8.44	0.255	8000	10	440.64	8812.8	0
10	10.6	8.7	0.255	8000	10	387.6	7752	12.03703704
20	10.6	8.95	0.255	8000	10	336.6	6732	23.61111111
30	10.6	9.2	0.255	8000	10	285.6	5712	35.18518519
40	10.6	9.41	0.255	8000	10	242.76	4855.2	44.90740741
50	10.6	9.6	0.255	8000	10	204	4080	53.7037037

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60	10.6	9.75	0.255	8000	10	173.4	3468	60.64814815
90	10.6	10	0.255	8000	10	122.4	2448	72.22222222
120	10.6	10.1	0.255	8000	10	102	2040	76.85185185

Table 10:- Sample (200ml) with 3 % activated carbon (6 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	%COD reduction
0	10.7	8.55	0.255	8000	10	438.6	8772	0
10	10.7	8.9	0.255	8000	10	367.2	7344	16.27907
20	10.7	9.3	0.255	8000	10	285.6	5712	34.88372
30	10.7	9.7	0.255	8000	10	204	4080	53.48837
40	10.7	10	0.255	8000	10	142.8	2856	67.44186
50	10.7	10.15	0.255	8000	10	112.2	2244	74.4186
60	10.7	10.27	0.255	8000	10	87.72	1754.4	80
90	10.7	10.4	0.255	8000	10	61.2	1224	86.04651
120	10.7	10.49	0.255	8000	10	42.84	856.8	90.23256

Table 11:- Sample (200ml) with 4 % activated carbon (8 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	%COD reduction
0	10.7	8.55	0.255	8000	10	438.6	8772	0
10	10.7	8.92	0.255	8000	10	363.12	7262.4	17.2093
20	10.7	9.35	0.255	8000	10	275.4	5508	37.2093
30	10.7	9.78	0.255	8000	10	187.68	3753.6	57.2093
40	10.7	10.1	0.255	8000	10	122.4	2448	72.09302
50	10.7	10.19	0.255	8000	10	104.04	2080.8	76.27907
60	10.7	10.3	0.255	8000	10	81.6	1632	81.39535
90	10.7	10.45	0.255	8000	10	51	1020	88.37209
120	10.7	10.53	0.255	8000	10	34.68	693.6	92.09302

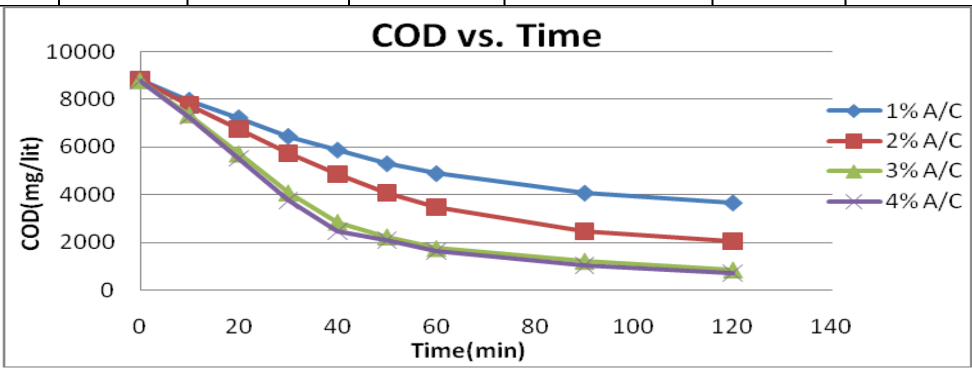

Fig 7

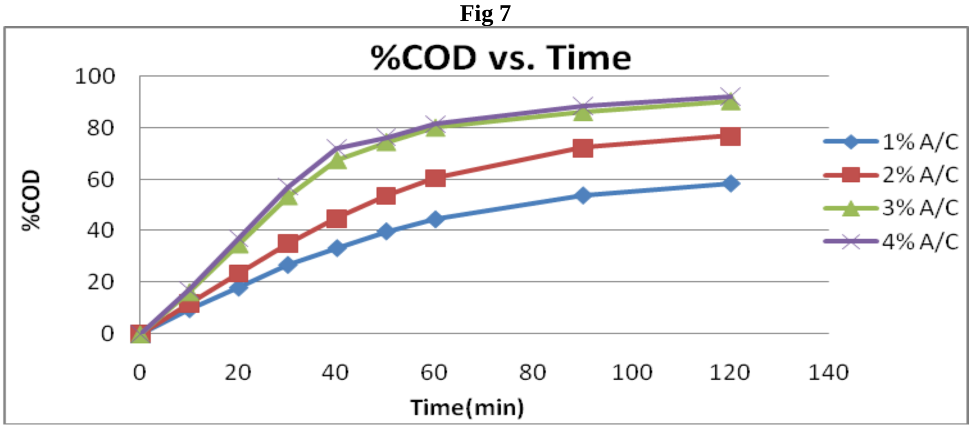

Fig 8

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Table 12:- Sample (200ml) with 1 % lignite powder (2 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	%COD reduction
0	11	8.87	0.255	8000	10	434.52	8690.4	0
10	11	9.1	0.255	8000	10	387.6	7752	10.79812207
20	11	9.35	0.255	8000	10	336.6	6732	22.53521127
30	11	9.6	0.255	8000	10	285.6	5712	34.27230047
40	11	9.79	0.255	8000	10	246.84	4936.8	43.19248826
50	11	10	0.255	8000	10	204	4080	53.05164319
60	11	10.14	0.255	8000	10	175.44	3508.8	59.62441315
90	11	10.37	0.255	8000	10	128.52	2570.4	70.42253521
120	11	10.45	0.255	8000	10	112.2	2244	74.17840376

Table 13:- Sample (200ml) with 2 % lignite powder (4 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	%COD reduction
0	11	8.87	0.255	8000	10	434.52	8690.4	0
10	11	9.15	0.255	8000	10	377.4	7548	13.14553991
20	11	9.4	0.255	8000	10	326.4	6528	24.88262911
30	11	9.63	0.255	8000	10	279.48	5589.6	35.68075117
40	11	9.85	0.255	8000	10	234.6	4692	46.00938967
50	11	10.05	0.255	8000	10	193.8	3876	55.39906103
60	11	10.2	0.255	8000	10	163.2	3264	62.44131455
90	11	10.52	0.255	8000	10	97.92	1958.4	77.46478873
120	11	10.65	0.255	8000	10	71.4	1428	83.56807512

Table 14:- Sample (200ml) with 3 % lignite powder (6 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	%COD reduction
0	10.7	8.59	0.255	8000	10	430.44	8608.8	0
10	10.7	8.97	0.255	8000	10	352.92	7058.4	18.00948
20	10.7	9.32	0.255	8000	10	281.52	5630.4	34.59716
30	10.7	9.63	0.255	8000	10	218.28	4365.6	49.2891
40	10.7	9.92	0.255	8000	10	159.12	3182.4	63.03318
50	10.7	10.2	0.255	8000	10	102	2040	76.30332
60	10.7	10.37	0.255	8000	10	67.32	1346.4	84.36019
90	10.7	10.5	0.255	8000	10	40.8	816	90.52133
120	10.7	10.55	0.255	8000	10	30.6	612	92.891

Table 15:- Sample (200ml) with 4 % lignite powder (8 gm)

Time (min)	Blank reading	Sample reading	Normality of FAS	MW of oxygen	Volume of sample	COD	COD*20	%COD reduction
0	10.7	8.59	0.255	8000	10	430.44	8608.8	0
10	10.7	9	0.255	8000	10	346.8	6936	19.43128
20	10.7	9.4	0.255	8000	10	265.2	5304	38.38863
30	10.7	9.7	0.255	8000	10	204	4080	52.60664
40	10.7	10	0.255	8000	10	142.8	2856	66.82464
50	10.7	10.3	0.255	8000	10	81.6	1632	81.04265
60	10.7	10.43	0.255	8000	10	55.08	1101.6	87.20379
90	10.7	10.52	0.255	8000	10	36.72	734.4	91.46919
120	10.7	10.58	0.255	8000	10	24.48	489.6	94.3128

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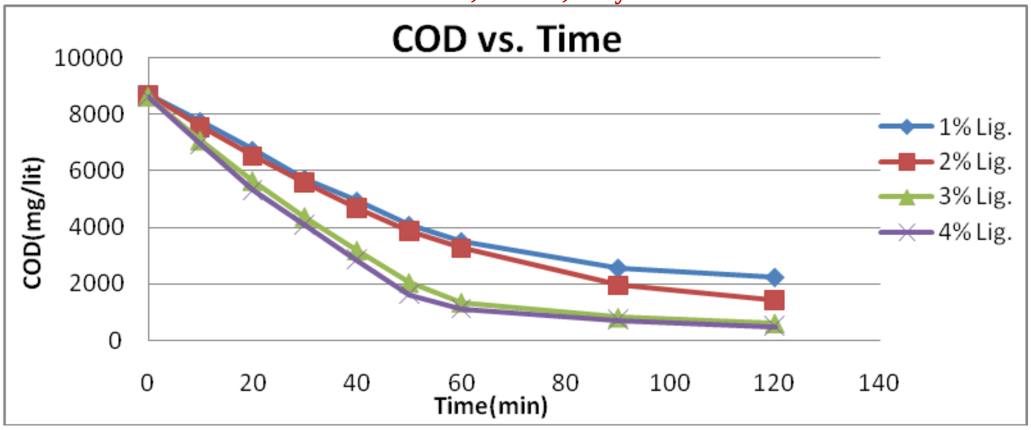

Fig 9

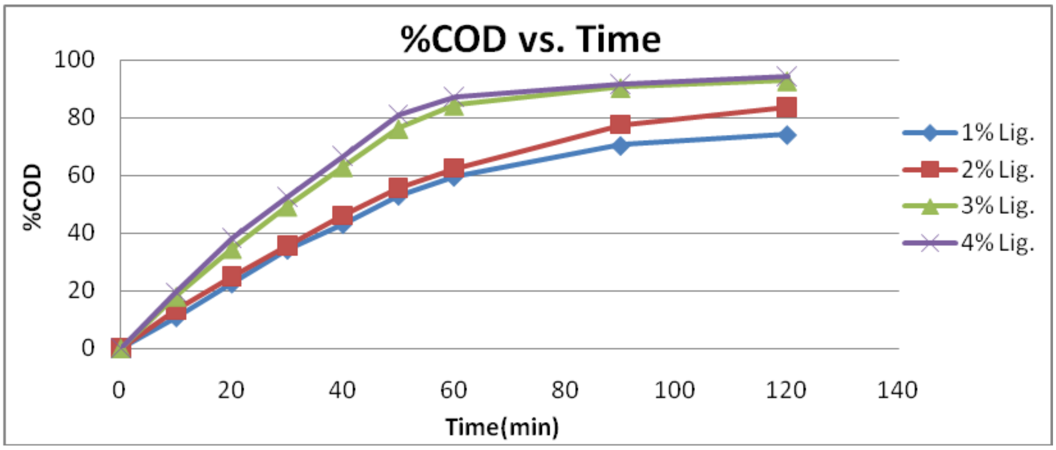

Fig 10

IV. CONCLUSION

It is clear from the above observations for the two cases under consideration that values of COD reduction for the case of acidic effluent are lower than the values of COD reduction for the case of neutral effluent. Moreover if the effluent is treated with lignite powder then COD reduction is higher than for treatment with activated carbon under identical conditions. Thus by modifying industrial effluent treatment flow sheet by carrying of adsorption operation initially than at the end, rates of COD can be decreased substantially to acceptable level. Hence, in industry, adsorption operation can be performed for desired contact time based on above data and corresponding rates of COD reduction for acidic effluent can be utilized conveniently to carry out "Effluent Treatment" more effectively. Thus, more quantity of effluent can be handled, or keeping effluent quantity same, effluent treatment can be carried for lesser contact time and operational cost can be decreased for the both categories.

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