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Charcoal-Based Effluent Pre-Treatment Strategy for the Solvent Production Plant of a Heavy Water Plant

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***Abstract*: This paper explores the application of activated charcoal adsorption for treating effluents generated from the Solvent Production Plant (SPP) of the Heavy Water Plant (HWP) in Tuticorin, India. The effluents generated from SPP contain organic solvents and hypersaline sodium chloride poses a significant challenge in terms of environmental compliance, specifically with the Tamil Nadu Pollution Control Board (TNPCB) prescribed limits for Chemical Oxygen Demand (COD). The methods of chemical effluent pre-treatments were technically cumbersome, unsustainable and not-ecofriendly since it needs multiple precipitations using dangerous chemicals to attain prescribed pollution control board limits and generates substantial solid waste. To address these limitations, the current study investigates the feasibility of charcoal adsorption as an alternative treatment method. The charcoal column setup, utilizing activated charcoal with a large surface area and a well-distributed pore structure, demonstrated a notable reduction in organic impurities, particularly in COD levels. Even though results show a significant decrease in COD of around 48%, the treated effluent still did not fully meet the TNPCB standards. The study also explores the regeneration potential of activated charcoal using a 1% HCl solution, which proved effective for reusing the charcoal. Linear regression and correlation coefficient analyses indicated a strong relationship between the volume of effluent and reductions in COD and Total Organic Carbon (TOC), emphasizing the potential of this method for large-scale industrial applications. This research suggests that activated charcoal adsorption could serve as an effective pre-treatment method for integrating in advanced Effluent Treatment System (ETS) at HWP Tuticorin.**

Keywords: Activated Charcoal, COD, Effluent Treatment, Regeneration, SPP

I. INTRODUCTION

The Solvent Production Plant (SPP) established in the Industrial premises of Heavy Water Plant (HWP) Tuticorin by the Heavy Water Board (HWB) a constituent unit of Department of Atomic Energy (DAE) engaged in the production of nuclear solvents required by the DAE for supporting the Indian Nuclear Power Program (INPP) along with heavy water production [1,2,3]. As per three stages of the nuclear power program, it involves various solvents used in the separation of valuable nuclear materials from front-end nuclear fuel cycle to back-end nuclear fuel cycle.

In HWP Tuticorin, solvent synthesis plant generates the effluents in aqueous solution form which contain sodium chloride contaminated with nuclear solvents and organic raw materials. The Chemical Oxygen Demand (COD) observed from the effluents varied in the range of 26,800 ppm, which is many times higher than the prescribed level of the state pollution control agency TNPCB. The limit prescribed by the TNPCB is in the range of less than 250 ppm of COD [5]. The pre-treatment studies with chemical-based method on treating effluents comes with limitations such as (a) not ecofriendly and unsustainable (b) Cost ineffectiveness. These challenges prompt the ecofriendly and sustainable approach involved to develop an alternate simplified Effluent Treatment System (ETS) for reducing the pollutant concentration exactly aligned with the TNPCB limits. Towards this, Charcoal adsorption method could be viewed as one of the pre-treatment options which may be integrated into a simplified energy efficient and sustainable effluent treatment system. In this pre-treatment method the effluent to be treated is passed through a column of charcoal where the impurities are trapped through the adsorption process of contaminants. The novelty of this study lies in applying a compact volume of 245 cm^3 activated - charcoal filter bed for the reduction of nuclear-solvent organic impurities and demonstrated a simple, sustainable regeneration method using 0.3 N HCl. This mild acid treatment effectively restores the adsorption capacity of the charcoal, enabling repeated reuse of the bed for organics removal. The combined approach offers a practical, low-cost, and resource-efficient treatment option for nuclear-solvent waste streams. As opined by many of the researchers, this technique could be considered as a sustainable alternate process to treat different kinds of effluents coming from domestic waste treatment plants [6]; mine industries [7]; food and pharmaceutical industries [8]; textile Industries [9,10,11]. The work presented here in this paper are related to the experimental research works carried out to know the efficacy of this charcoal adsorption technique in treating the effluents coming from the SPP unit of HWP.

II. MATERIALS AND METHODS

To comply with environmental standards, the physicochemical characterization of the effluents is one of the basic needs for effective monitoring of effluent discharge. Experimental analysis of real-time effluent samples provides precise solutions for addressing site-specific industrial wastewater challenges. The investigations in the present study were carried out in the Process Control and Analytical (PC & AL) Laboratory of HWP Tuticorin, India. The required effluent samples were collected from the SPP of HWP Tuticorin. The physicochemical properties of the effluent were measured following standard procedures outlined in the water quality analysis protocol of HWP Tuticorin. Table 1 gives the information on the various physico-chemical parameters that were measured, the instruments used, the specifications of these instruments, along with the TNPCB limits.

TABLE 1
Details of physico-chemical parameters measured, instruments used and TNPCB limits

Parameter	Instrument used	Range of the Instrument	TNPCB Limit
Density (g/cc)	Mettler Toledo – weighing machine	0.0001 g to 180g	----
pH	pH machine – Labindia PICO +	1-14	6.5 – 8.5
Conductivity, k (mS/cm)	Conductivity meter – Labindia PICO+	0.001 µS/cm-100ms/cm	2.25 mS/cm
TDS (ppm)	Gravimetric Method	0-1000 ppm	2100 ppm
Na^+ (%)	Flame Photometry Method	0-100 ppm	60 %
Cl^- (%)	Argentometric Titration Method	0.15 – 10mg	0.06 %
COD (ppm)	Redox Titration Method	50-800 ppm	< 250 ppm

Adsorption mechanism in Charcoal column

The solvents in SPP are synthesized based on organophosphorus compounds and organoamide based compounds. The adsorption capacity of activated charcoal was highly effective in reducing organic impurities, owing to its numerous active adsorption sites. The efficiency of activated charcoal is attributed to its well-distributed pore structure (Figure 1), which includes macropores (d > 50 nm), mesopores (2 nm < d < 50 nm), and micropores (d < 2 nm).

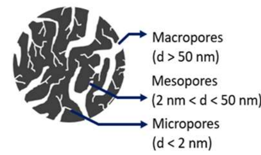


Fig.1: Distribution of pores in Activated Charcoal [12]

Adsorption occurs within and on these pores, trapping macromolecular organic impurities and significantly reducing contaminant levels through sustainable filtration. Activated carbon features numerous micropores and has a surface area exceeding 1,500 m^2/g, allowing it to effectively remove color, odor, organic and inorganic pollutants, heavy metals [13]. The mechanical strength of activated carbon is a key characteristic that defines its quality and durability, as it determines the material's ability to withstand wear and tear [14].

The charcoal filter bed adsorption process intercepts pollutants in suspension with organic matter and contaminants filling the gaps between activated carbon particles. The pore size and porosity of the charcoal filter bed increase with larger particle sizes of activated carbon. The experimental observation in the charcoal column exhibits potential filtration effects of charcoal filter bed granules which facilitates deeper entrapment of suspended organic impurities within the porous zones.

Design of Charcoal Column

An experimental setup with a charcoal column of 2 feet (60 cm) in height and 1 inch (2.5 cm) in internal diameter has been designed. Figure 2 depicts the design features of this column. The column has an inbuilt perforated disc with 1 mm holes at the column's end before the control valve, facilitating the preparation of the charcoal bed. The calculated technical specifications for the design of the charcoal exchange column are as given in the table 2. As observed from figure 2 and table 2, a charcoal column volume of 294.375 cm^3 was required to treat a 250 mL sample of real-time effluent. On an industrial scale, treating 7 m^3 of effluent from the SPP unit would require a charcoal exchange column with a holding capacity of 8,24,250 cm^3. Similarly, 6,868 kg of charcoal is needed to treat the 7 m^3 of SPP effluents.

TABLE 2
Design specifications of the charcoal column

Technical properties of charcoal and column	Quantity measured
Material, Length and Diameter	Stainless Steel, 2feet, 1 inch
Bulk density of activated charcoal (granular)	40g / 100 ml
Free space in the fully loaded charcoal column	49 cm^3
Total volume of charcoal exchange column	294 cm^3
Volume of charcoal filter bed in the column	245 cm^3
Activated charcoal for filter bed	98 g

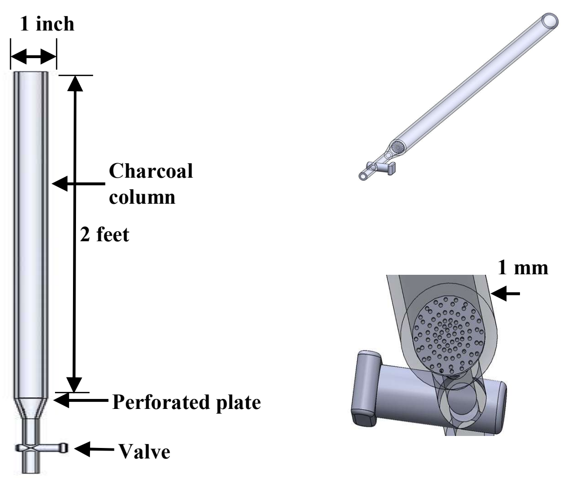


Fig.2: Schematic of Charcoal Exchange Column (with isometric view and enlarged view of perforated plate)

III. Results and Discussion

Real-time effluents from SPP at HWP Tuticorin were analyzed in the Process Control & Analytical Laboratory, and the performance of charcoal adsorption was evaluated by comparing feed and treated samples. Activated carbon showed strong adsorption characteristics [15], and treatment effectiveness was assessed using TOC and COD measurements across multiple sampling stages. Linear regression and correlation analyses were used to examine data trends [16]. Regeneration studies further confirmed the reliability and sustainability of the charcoal bed for effluent treatment.

Characteristics of effluents before and after treatment

Table 3 presents the effluent characteristics of the SPP feed effluent, analyzed in the Process Control & Analytical Laboratory of the SPP unit at HWP Tuticorin. The effluent was within permissible limits after neutralization, consistent with standard industrial practices [17]. However, the elevated values of other parameters indicate the need for further treatment to meet the discharge standards prescribed by TNPCB and CPCB [18].

Table 4 presents the effluent characteristics of the SPP effluent after undergoing charcoal adsorption treatment. The results indicate a notable reduction in Chemical Oxygen Demand (COD), reflecting the effective removal of organic impurities which is consistent with the adsorption behavior typically reported for activated adsorption systems [17]. However, parameters such as conductivity (K), Total Dissolved Solids (TDS), sodium ion, and chloride ion showed no significant reduction, as their measured values remained consistent with those of the feed effluent. This observation aligns with established findings that activated carbon primarily removes organic and certain inorganic species but has limited capacity to reduce dissolved ionic constituents [18]. Additionally, a rise in pH levels was observed, likely due to chemical interactions between the effluent's composition and the activated carbon filter bed, a phenomenon also documented in standard water treatment literature [18, 19].

TABLE 3
Characteristics of Effluents (before charcoal treatment)

Mixture of effluents from Solvent	Density (g/cc)	pH	Conductivity K (mS/cm)	TDS (ppm)	Na^+ (%)	Cl^- (%)	COD (ppm)
TBP, TIAP, TOPO, DHOA, D_2EHPA -II	1.037	6.0	43	28,681	0.98	1.5	26,800

TABLE 4
Characteristics of an Effluents (after Charcoal treatment)

Mixture of effluents from Solvent	Density (g/cc)	pH	Conductivity K (mS/cm)	TDS (ppm)	Na^+ (%)	Cl^- (%)	COD (ppm)
TBP, TIAP, TOPO, DHOA, D_2EHPA -II	1.0085	8.09	44.71	28,020	0.91	1.48	13790

COD Analysis

COD represents the amount of oxygen required to chemically oxidize organic matter in SPP effluents and is a key indicator of organic pollution in industrial wastewater [20]. Higher COD value reflects increased concentrations of oxidizable substances (organics), which can lead to severe oxygen depletion in receiving water bodies. In this study 5,000 mL of effluent was analyzed, of which 4,750 mL was passed through a charcoal adsorption column in 19 stages at 250 mL per stage.

Continuous monitoring of COD until equilibrium was observed (Fig.3) enabled determination of overall average COD concentration [21].

Stage	Volume (ml)	COD (ppm)
1	250	1,200
2	500	3,600
3	750	4,794
4	1000	6,000
5	1250	7,200
6	1500	10,800
7	1750	11,025
8	2000	11,600
9	2250	12,217
10	2500	12,800
11	2750	13,150
12	3000	13,600
13	3250	17,375
14	3500	18,633
15	3750	19,892
16	4000	22,316
17	4250	23,555
18	4500	26,082
19	4750	26,182

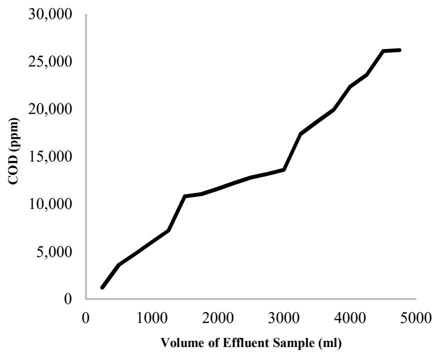


Fig.3: Variation of COD level in different stages of effluent samples

Total Organic Carbon (TOC) Analysis

In SPP effluents, organic contamination arises mainly from raw materials and by-products formed during solvent synthesis, including organophosphorus compounds containing C-P bonds. The high COD reflects a significant load of suspended and dissolved organic impurities, while TOC quantifies the overall carbon content and indicates the degree of contamination. After passing the effluent through an activated charcoal column, the reduction in TOC between the feed and the treated samples represents the carbon adsorbed calculated as:

Reduction in TOC = TOC of feed effluent – TOC of treated effluent ..Eq (a)

In this study, the feed effluent showed a TOC of 1,675 mg. A total of 4,750 mL was processed through a charcoal column in multiple stages (19 stages × 250 mL), and TOC measured at each stage until equilibrium was reached, allowing average estimation of average TOC removal and adsorption efficiency. The use of activated carbon for removing organic pollutants including solvents, phenolics, and other industrial organics – is well established due to its high surface and strong adsorption capacity [22,23].

Stage	Volume (ml)	TOC (mg)	‘C’ adsorbed (mg)
1	250	75	1,600
2	250	225	1,450
3	250	300	1,375
4	250	375	1,300
5	250	450	1,225
6	250	675	1,000
7	250	700	975
8	250	725	950
9	250	775	900
10	250	800	875
11	250	825	850
12	250	850	825
13	250	975	700
14	250	1,175	500
15	250	1,200	475
16	250	1,250	425
17	250	1,300	375
18	250	1,630	44.9
19	250	1,636	38.625

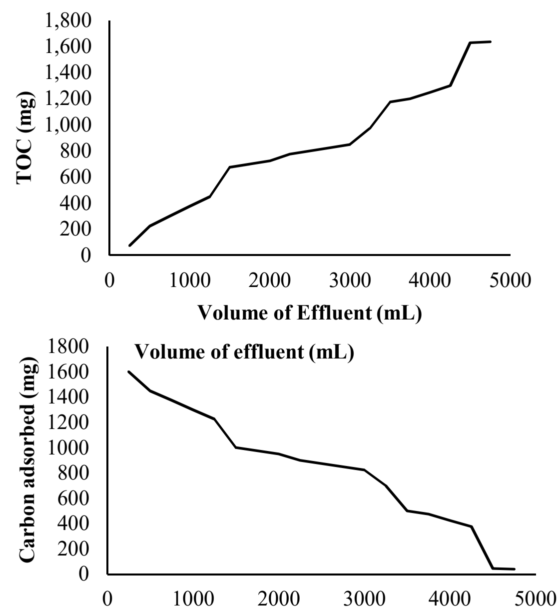


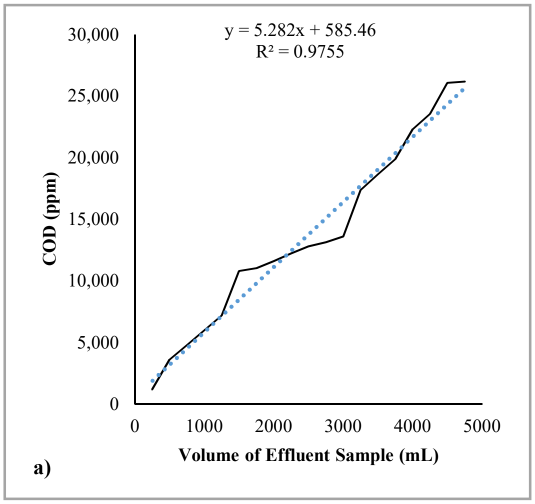
Fig.4: Variation of TOC and Carbon adsorbed in different stages of effluent samples in activated charcoal filter bed

Linear Regression and Correlation co-efficient Analysis and resultant equilibrium equation

A linear regression analysis was performed between the volume of effluent treated and the corresponding COD (and TOC) values. The resulting regression equation (with non-zero intercept) represents the equilibrium behavior of the charcoal effluent treatment system; the high co-efficient of determination ($R^2 > 0.95$) confirms a very strong linear relationship between treated volume and COD/TOC response. In the case shown in figure 5a, the slope is 5.282 – meaning every additional 1mL of effluent treated corresponds to an increase of 5.28 ppm in COD. This indicates that as more wastewater is processed, the oxygen demand as (COD) increases, implying discharge of organic pollutants.

Similar linear correlations between COD and TOC (or other treatment metrics) have been demonstrated in industrial wastewater studies, allowing use of regression models for prediction and monitoring studies using activated carbon for COD and TOC removal [24].

In Figure 5b, the slope of 0.3117 indicates that for every 1 mL increase in treated effluent volume, TOC increases by approximately 0.31 mg. This positive slope shows a direct relationship, where increasing treated volume corresponds to rising TOC levels. The solid line represents the measured data, and the dotted line shows the linear-regression trend. Similar linear correlations between TOC and treatment parameters have been widely reported in industrial wastewater studies.



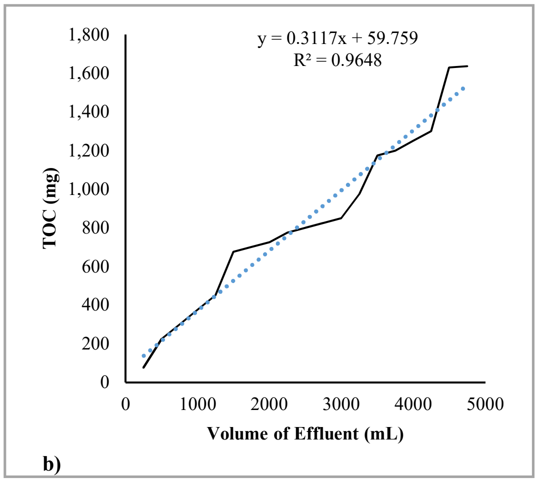


Fig.5 (a & b): Linear Regression and Correlation Coefficient Analysis of COD and TOC vs Effluent volume

In this statistical analysis, linear regression provides a structured approach to derive equilibrium equations in water treatment [25,26], while the correlation coefficient (R^2) ensures model validity [27]. These statistical tools help to optimize treatment processes by accurately describing contaminant interactions, leading to better water purification system design and operation [28]. In charcoal adsorption effluent treatment method these statistical tools optimizing adsorption efficiency, predicting treatment outcomes, and improving water purification processes.

Regeneration of Activated Charcoal

A total of nineteen samples, each measuring 250 mL out of 4750 mL, were analyzed for charcoal adsorption in a charcoal exchange column. A sieved (1.68 mm pore size) quantity of 98.12 g of activated charcoal was introduced into a 2-foot-high charcoal column with a total volume of 294 cm³. As the effluent samples passed through the charcoal filter bed, a significant reduction in organic pollutants was observed, along with noticeable color removal, consistent with typical fixed bed activated carbon performance reported in literature [29]. Breakthrough occurred in the initial stages of filtration, while saturation was reached at the eighteenth and nineteenth stages, which aligns with classic breakthrough – exhaustion behavior in adsorption columns

To enable the reuse of the activated charcoal filter bed, regeneration studies were conducted using a 1% HCl solution (0.3 N). Acid regeneration of exhausted activated carbon is well documented as an effective method for restoring adsorption capacity [30]. The acid treatment proved effective when applied after the equilibrium stage (saturation). Following regeneration, a 250 mL effluent sample (feed) was tested through the charcoal column, showing a COD reduction equivalent to that observed in the initial treatment stage, consistent with reports of successful chemical regeneration restoring fresh-carbon performance [31]. The regeneration process with 1% HCl was more effective in higher pH buffered complexes compared to low pH buffered complex adsorption on the charcoal bed, which aligns with established pH-dependent adsorption and desorption behavior of organic compounds [32].

IV. Conclusion

The activated charcoal adsorption effluent treatment method effectively reduces organic pollutants in the Solvent Production Plant (SPP) of Heavy Water Plant (HWP) Tuticorin. The high adsorption capacity of activated charcoal, attributed to its large surface area and well-distributed macro, meso, and micropores, enhances the filtration of suspended impurities in the charcoal exchange column. The treated effluent did not fully meet Tamil Nadu Pollution Control Board (TNPCB) statutory limits, the significant reduction in Chemical Oxygen Demand (COD) to 13,790 ppm from 26,800 ppm highlights its potential as a viable pre-treatment method for an Integrated Effluent Treatment System (IETS) on an industrial scale. The breakthrough adsorption behavior and regeneration potential of activated charcoal reinforce its sustainability and industrial applicability. Linear Regression and Correlation Coefficient analysis of COD and Total Organic Carbon (TOC) confirm a strong model fit, with an R^2 value classified as excellent, indicating a strong correlation between COD and TOC trends across varying volumes. These findings underscore the importance of developing a sustainable, techno-economically viable pre-treatment approach for the IETS of SPP at HWP Tuticorin.

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