

Research Article

# Effective Treatment of Acidic Effluent Generated from Li-ion Battery Recycling

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## Abstract

The present study investigates a comprehensive treatment strategy for managing acidic effluent generated during the hydrometallurgical processing of discarded lithium-ion batteries (LIBs), specifically following cobalt oxalate precipitation. The effluent, characterized by extremely low pH (0.1), high total dissolved solids (TDS = 50,000 mg/L), and elevated chemical oxygen demand (COD = 1640 mg/L), was treated through a sequential combination of coagulation, adsorption, and distillation. Coagulation using ferric sulfate achieved 34% TDS reduction through precipitation of dissolved metal ions and oxalates. Subsequent adsorption employing thermally activated carbon derived from waste RO filters further reduced TDS by ~55% due to enhanced surface area and porous structure. Final distillation at 150 °C yielded a >99% decrease in TDS and COD, producing condensate meeting CPCB discharge standards (TDS = 79 mg/L, COD = 32 mg/L). The integrated approach effectively transformed a high-strength acidic effluent into reusable water while concentrating recoverable metal residues. A preliminary techno-economic assessment indicated that the process is technically viable and scalable, with energy consumption during distillation being the major cost factor. The study demonstrates a sustainable and resource-efficient treatment pathway for LIB recycling effluents, contributing toward circular economy and zero-liquid discharge objectives.

## Keywords

Effluent Treatment, LIBs Recycling, Coagulation, Adsorption, Distillation

## 1. Introduction

Global environmental issues like the energy crisis and climate change can be addressed by developing sustainable energy solutions and implementing circular economy practices. For the past decade, the key focus has been shifted to decarbonizing the transport sector by adopting electric vehicles (EVs) which could reduce greenhouse gas emissions. EV adoption accelerates the demand for lithium-ion batteries (LIBs) as the primary source for electric propulsion and stationary energy storage applications [1]. LIBs have revolu-

tionized our everyday lives with their potential use as a power source for consumer electronics and stationary energy storage devices. LIBs are widely accepted due to their high energy density, high voltage operation, extended lifespan, lower self-discharge, broad working temperature range, compact size and weight, and excellent electrochemical features [2, 3]. LIBs lifespan ranges from 10 to 15 years [4]. The global demand for lithium-ion batteries is projected to increase almost seven-fold between 2022 and 2030, reaching 4.7

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terawatt-hours in 2030 [5].

Due to significant growth in the demand for LIBs for their potential use as an energy source, there is a huge requirement for starting precursor materials for battery manufacturing. The important raw materials required for the synthesis of battery cathode materials are cobalt (Co), lithium (Li), manganese (Mn), and nickel (Ni). Global reserves for critical materials are limited, with the Democratic Republic of Congo (DRC) producing ~73.3% of Co, Australia, and Chile producing ~80% of Li, and Indonesia and the Philippines producing ~51% of Ni demand worldwide [6]. Recycling discarded LIBs at their end-of-life has been a viable option to recover valuable materials for their potential use as secondary raw materials in battery manufacturing to meet the demand and mitigate the foreign dependency for starting/critical materials [7].

Both pyrometallurgical as well as hydrometallurgical routes are applied for the recovery of metals/materials from discarded end-of-life LIBs [8]. Pyrometallurgy processing involves high-temperature smelting followed by separation steps for recovery of metals alloys [9, 10], whereas hydrometallurgical processing involves acid leaching in chemicals followed by selective extraction of metal contents through solvent extraction, precipitation, etc. [11]. The pyrometallurgical route is an energy-expensive route applied for recovery of metal alloy as a product and generates flue gasses and slag [12]. Hydrometallurgy processing remains a preferred choice for selective extraction of metal contents present in the batteries which can further be used as secondary raw materials for battery manufacturing [13]. Nevertheless, the hydrometallurgical methodology produces a significant quantity of effluents that necessitate efficient treatment prior to their ultimate discharge. The release of effluents must adhere to the discharge limits prescribed by the regulatory authorities.

The effluent composition depends upon the process flow and associated chemistry of extractants. There are reports on the treatment of effluents that apply different methodologies such as adsorption [14-16], coagulation [17, 18], a combination of coagulation and adsorption [19]. The most common synthetic coagulants used in coagulation processing are poly (aluminum chloride), poly (aluminum sulfate chloride) and poly (aluminum silicate chloride) [20]. Coagulation process efficiency is influenced by temperature, pH, effluent quality, concentration, and type of coagulant in the coagulation-flocculation process [21, 22]. The coagulation process has widespread application in effluent treatment due to its process simplicity and cost-effectiveness [23, 24]. The jar test is the most common technique for evaluating and optimizing the coagulation-flocculation processes. The test comprises simultaneous and consecutive sedimentation, and fast, and slow mixing processes. The jar test is a pilot-scale assessment of the treatment. It mimics the mechanisms of flocculation and coagulation found in effluent treatment facilities. It determines the proper dosages of treatment chemicals, and the efficiency of the plant [25].

Adsorption is a tertiary effluent treatment process that in-

volves the removal of contaminants from wastewater through their adherence to the surface of an adsorbent material. Activated carbon is the most common adsorbent due to its high surface area and porous structure. Activated carbon adsorption offers the advantage of removing a wide range of compounds, easy and flexible accessibility, and low risk of toxic byproduct generation [26, 27]. The Multi-effect evaporation (MEE) studies for the treatment of different effluents have also been reported in the literature which includes additional processing steps such as evaporation, distillation, column stripping, etc. [28-30]. Evaporation is a thermal process for liquid removal from a solution by boiling off a portion of the liquid, resulting in a more concentrated product [31]. The process solution is introduced followed by heat treatment. Simultaneous evaporation and condensation of liquid results in concentrated solution for further processing [32]. The distillation process is a lab-scale experiment model of a multi-effect evaporation (MEE) process. In this process in wastewater treatment include solvent recovery, salt removal from water, and the removal of volatile organic compounds (VOCs).

Effluent generated from hydrometallurgical processing of spent lithium-ion batteries (LIBs) contains high concentrations of dissolved metal ions, oxalic acid residues, and other toxic compounds, posing severe environmental hazards if discharged untreated. Such effluents often exhibit extreme parameters: high chemical oxygen demand (COD), very high total dissolved solids (TDS), and low pH, making treatment challenging. Conventional methods—coagulation, adsorption, membrane filtration, or combinations thereof—frequently yield partial pollutant removal for such demanding waste streams [33, 34]. For instance, in distillery wastewater treated with nanofiltration (NF) and reverse osmosis (RO), Wang et al. reported ~99.80% TDS removal and ~99.90% COD reduction, though with high-pressure operation and high capital/operating cost [33]. Meshram, Thakur & Soni (2021) investigated Pb(II) removal from battery recycling effluent using steam-activated granular carbon and achieved over 95% Pb(II) removal under optimized conditions [34]. Meanwhile, other studies of adsorption from sewage or industrial effluents (e.g. date palm-shell derived carbon) have achieved ~95% COD removal but typically at lower TDS and lower acidity levels than those encountered in LIB recycle acidic effluents (e.g., date-palm waste AC: ~95.4% removal of COD at moderate conditions) [35]. Despite these successes, achieving >99% removal of TDS and COD from effluent streams that are both highly acidic and heavily laden with oxalate and cobalt remains rare. Many prior reports either (a) deal with more benign effluents, (b) do not include a distillation or equivalent final purification step, or (c) do not treat the combined challenge of high dissolved solids and acid organics simultaneously.

In prior investigations, the authors have reported on the processing of discarded LIBs through both hydrometallurgical as well as pyro metallurgical routes [36-39]. In this work,

an integrated treatment scheme in sequential steps of coagulation, adsorption and distillation is applied to an oxalic acid-based effluent from cobalt oxalate precipitation, with initial conditions of pH  $\approx$  0.1, TDS  $\approx$  50,000 mg/L, and COD  $\approx$  1640 mg/L. Under these extreme conditions, the process achieved >99% removal of both TDS and COD, producing a condensate with TDS  $\sim$ 79 mg/L and COD  $\sim$ 32 mg/L, fully compliant with Centre Pollution Control Board (CPCB) (India) discharge norms. These results represent a significant technical advances, particularly in treating highly acidic, metal-oxalate laden effluents under near-real recycling plant conditions.

## 2. Materials and Methodology

### 2.1. Process Instruments, Materials, and Reagents

Acidic effluent is generated during the processing of discarded LIBs while selective extraction of Co content through hydrometallurgical route; this effluent is subsequently collected for the purposes of the current investigation. The physical and chemical parameters of the as-received effluent were measured before and after the treatment processes. The effluent quality parameters were analyzed for APHA standards compliance (American Public Health Association, 1999) [40]. pH was measured by using the Digisun Electronics System (Model 2001). TDS and conductivity values of the studied effluent were measured by using the Eutech Instruments (Model PC650). The TDS refers to the combined content of all inorganic salts and small amounts of organic matter present in the effluent liquid, which represents the sum of cations (e.g., calcium, magnesium, sodium, potassium) and anions (e.g., bicarbonates, chlorides, sulfates, nitrates) dissolved in the water. TDS provides a measure of the overall salinity or ionic strength of the effluent. In this study, the total dissolved solid quantity specifically denotes the concentration

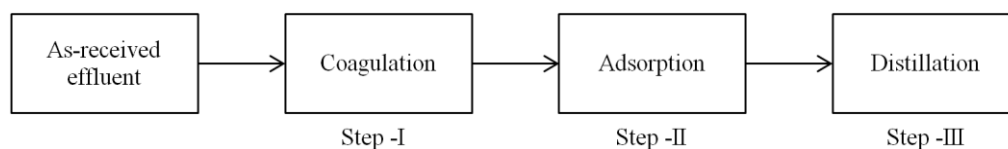
of dissolved substances per unit volume of effluent, measured as mg/L, in accordance with APHA Standard Methods (2540 C). Stirring experiments were carried out using an IKA CMAG HS7 digital hot plate magnetic stirrer. Heat treatment of samples was carried out using a muffle furnace. The concentration of metallic elements present in the studied material at various processing steps was analysed by using inductively coupled plasma-optical emission spectrometry (ICP-OES; 720 Agilent). Aluminum sulfate ( $\text{Al}_2\text{SO}_4 \cdot 16\text{H}_2\text{O}$ , 98 wt.%), Ferric sulfate hydrate ( $\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$ ), Aluminum oxide ( $\text{Al}_2\text{O}_3$ ) were chemicals and reagents used in different stages of effluent treatment. pH values of samples were adjusted using sodium hydroxide (NaOH, 99.4wt%) solution. The COD value of the effluent before and after treatment was measured through a National Accreditation Board for Testing and Calibration Laboratories (NABL) laboratory in India.

### 2.2. Sample Analysis

Total suspended solids (TSS), total dissolved solids (TDS), electrical conductivity, pH, temperature, chemical oxygen demand (COD), and biochemical oxygen demand (BOD) are effluent quality parameters considered in the present study to reduce the permissible limit set by CPCB [41]. Table 1 illustrates the various effluent quality parameters obtained for as-received acidic effluent. The effluent discharge limits set by CPCB; in India are also mentioned in Table 1. The effluent sample showed a high COD value (1640 mg/L) while the BOD was reported as ND (Not Detected). This observation is consistent with the nature of the effluent, which exhibited extremely high TDS (50,000 mg/L), very low pH (0.1), and elevated conductivity (75 mS). Such conditions are inhibitory to microbial activity, thereby preventing biodegradation during the BOD test. A highly acidic pH (0.1) that is lethal to microbes. In addition, the effluent is likely to contain non-biodegradable or toxic organics, which contribute to COD but do not support microbial oxidation. Hence, the BOD was observed as ND despite a high COD level.

**Table 1.** Quality parameters of as-received acidic effluent and their limit for discharge set by CPCB India.

| Effluent quality parameter   | Unit | Method                                     | As-received effluent | Discharge limits (as per CPCB, India) |
|------------------------------|------|--------------------------------------------|----------------------|---------------------------------------|
| Total suspended solids (TSS) | mg/L | APHA 23 <sup>rd</sup> Edition 2540 D       | 20,000               | <100                                  |
| Total dissolved solids (TDS) | mg/L | APHA 23 <sup>rd</sup> Edition 2540 C       | 50,000               | <2100                                 |
| Electrical conductivity      | mS   | APHA 23 <sup>rd</sup> Edition 2510 B       | 75.00                | --                                    |
| pH                           | --   | APHA 23 <sup>rd</sup> Edition 4500 H+B     | 0.1                  | 5.5-9.0                               |
| Temperature                  | °C   |                                            | 25                   | within 5 °C of water body temperature |
| Chemical oxygen demand       | mg/L | APHA 23 <sup>rd</sup> Edition 2012, 5220 B | 1640                 | <250                                  |
| Biochemical oxygen demand    | mg/L | IS 3025 Method                             | ND                   | <100                                  |



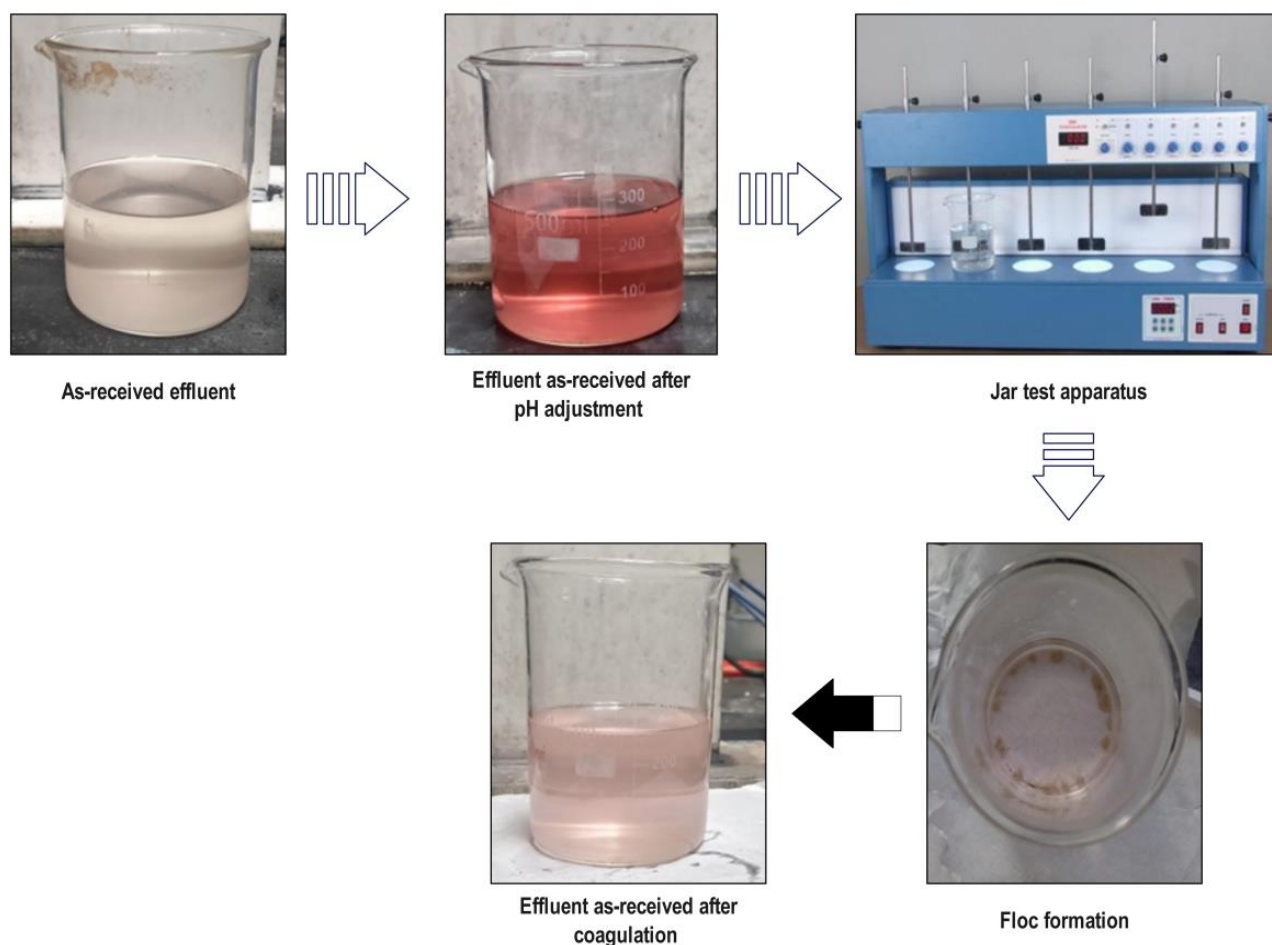
**Figure 1.** The process flow applied in the current study for effective effluent treatment.

Figure 1 shows the process flow applied in the current study for effective treatment of as-received effluent. The processing involves coagulation followed by adsorption and then distillation to effectively treat the as-received acidic effluent.

### 2.3. Coagulation

The coagulation process involves floc formation, particle destabilization, and interparticle collision. Aluminium sulfate and ferric sulfate are used as coagulants in the current study and results were compared. The pH value of as-received effluent is found between 0 to 1pH. The pH operating range of aluminum sulfate and ferric sulfate ranges between 5.5 to 7.0 and 4.5 to 6.0, respectively. The effluent pH adjustment was carried out using sodium hydroxide (NaOH). During cobalt oxalate precipitation, not all cobalt is removed; some  $\text{Co}^{2+}$  ions remain dissolved in the oxalic acid mother liquor. The

red coloration observed upon pH adjustment of the oxalic acid-based effluent arises due to the hydrolysis and complexation of residual cobalt ions. At higher pH, dissolved  $\text{Co}^{2+}$  forms cobalt-oxalate complexes and hydroxide precipitates, which exhibit characteristic red to pink coloration. This indicates that some amount of cobalt species remain present in the effluent even after the primary precipitation step. Coagulation experiments were carried out in a jar test apparatus. During the jar test, rapid mixing at 100 rpm for 1 minute followed by slow mixing for 20 minutes was conducted. A settling time of 30 minutes was provided for floc formation and settling. A batch of six effluents was carried out. The batch size of the coagulation process was 500 mL. The coagulant concentration was varied from 0.5 mg/L to 10 mg/L. The process flow of the coagulation process is displayed in Figure 2.



**Figure 2.** The process flow of the coagulation process applied for effluent treatment.

## 2.4. Adsorption

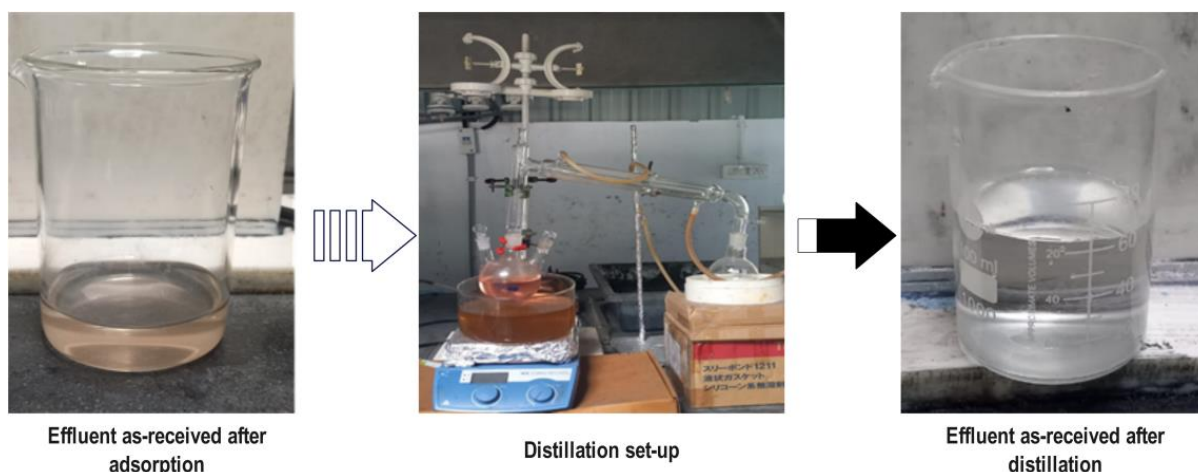
A fixed bed multilayer batch adsorption process was carried out. The activated carbon from the waste cartridge of the RO filter system was recovered and used as adsorbent in the present study. The activated carbon powder obtained after crushing and grinding the waste RO filter cartridge was subjected to thermal activation at 400 °C for 1 h in a muffle furnace in ambient atmosphere. This treatment primarily served to remove moisture, volatile organics, and surface impurities, thereby exposing previously blocked adsorption sites. At this moderate activation temperature, significant pore cleaning and reopening of micro- and mesopores is expected, without extensive structural collapse that may occur at temperatures higher than 400 °C. As a result, the thermally treated carbon likely exhibits an increase in effective surface area and pore accessibility, which directly enhances adsorption performance. Moreover, the heat treatment alters the surface functional groups, reducing unstable oxygenated groups and creating a cleaner carbonaceous matrix with improved affinity for pollutant molecules. Thus, thermal activation at 400 °C provides an optimal balance between surface area enhancement and structural stability, making the recovered RO filter-derived activated carbon a promising low-cost adsorbent for effluent treatment applications. An adsorption column is prepared with alternate multilayer stacking of activated carbon and aluminium oxide. The bottom of filter bed was filled with glass wool to provide support to the filter bed at the bottom open end of the adsorption column. The thickness of one layer (aluminium oxide and activated carbon) is maintained constant at ~5cm. A 7-layer adsorption column was prepared for the adsorption processing of effluent. Figure 3 shows the image of the adsorption column prepared for the current study. The as-received effluent after coagulation is allowed to pass through the prepared 7-layer adsorption column. Continuous monitoring of TDS, electrical conductivity, and pH was carried out throughout the experiment.



**Figure 3.** Adsorption column prepared using multilayer stacking of activated carbon and aluminium oxide.

## 2.5. Multi-Effect Evaporator (MEE)

In the present study, distillation was carried out at normal atmospheric conditions. Figure 4 shows the process flow used in the distillation of effluent in the present study. The effluent obtained after adsorption was processed in a distillation set-up to reduce the TDS concentrations. Steam was generated at temperature of 150 °C and 160 °C which was then condensed and collected at room temperature. In the current study, lab-scale experiments were carried out using a 3-three neck round bottom (RB) flask as an evaporator. The RB flask was immersed in an oil bath for uniform heating. The evaporator was connected to a condenser and collector for the collection of distilled effluent. A clear effluent solution was obtained, which was further analysed for TDS and electrical conductivity values. The solid concentrates were collected as residue after distillation from the evaporator.



**Figure 4.** The process flow applied for distillation step in effluent treatment.

### 3. Results and Discussion

#### 3.1. Coagulation Process

Table 2 shows the ICP-OES results for analyzing elemental metal concentration in the studied effluent generated from recycling discarded LIBs through the hydrometallurgical route. The major metal concentrations in the LIBs such as Li, Co, Ni, and Mn were analyzed for their presence in the studied effluent. The studied effluent was generated from the processing of batteries for selective extraction of Co values, which is acidic. It

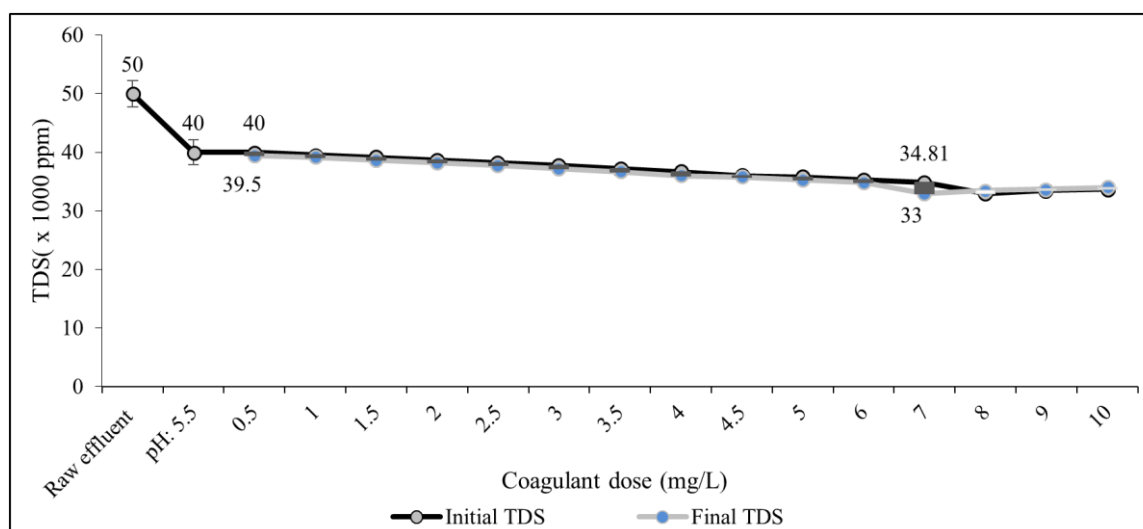
is found that the quality parameters observed for studied effluent including pH, TDS, TSS, COD, and BOD are above the permissible limit to discharge set by CPCB India (Table 1). The high TDS value found in as-received effluent is due to the presence of dissolved concentrations as illustrated in Table 2. A comparative coagulation process of two coagulants (ferric sulfate and aluminum sulfate) was conducted for acidic effluent [42]. Coagulants form a gelatinous gel-like surface, suspended particles are attracted to its surface. During the coagulation process, floc formation takes place.

**Table 2.** ICP-OES results of effluent generated from LIB recycling.

| Sample                                  | Results in wt. % |       |    |    |       |       |    |       |
|-----------------------------------------|------------------|-------|----|----|-------|-------|----|-------|
|                                         | Al               | Co    | Cu | Fe | Li    | Mn    | Ni | P     |
| As-received effluent from LIB recycling | 0.003            | 0.005 | ND | ND | 0.002 | 0.008 | ND | 0.008 |

The curves shown in Figure 5 represent a change in TDS during the coagulation process by using aluminium sulfate as a coagulant [43-45]. Lithium-ion battery recycling process through hydrometallurgy route is carried out through high concentration of acid leaching process. High-concentration acid leaching process reduces the pH of the effluent to below 1 pH level. pH adjustment is carried out for the optimum TDS removal from effluent. The initial TDS of effluent is 50000 ppm which decreases to 40000 ppm after pH adjustment to 5.5 [46]. The metal concentrates present in the acidic effluents react with NaOH (added during pH adjustment) and forms hydroxides which reduces the TDS concentration [47]. It is

found that the final TDS value decreases from 39500 ppm to 33000 ppm with an increase in coagulant dose from 0.5 mg/L to 7 mg/L, respectively. Further addition of coagulant dose results in an increase of TDS concentration in the effluent. Optimization of coagulant dosage is important to obtain maximum TDS removal efficiency. Maximum TDS removal efficiency was obtained at 7 mg/L coagulant dosage. Using aluminum sulfate coagulant, 34% TDS reduction efficiency is obtained during coagulation. Coagulant dosage above 7 mg/L will re-suspend settled dissolved and suspended solids into the effluent, which eventually increases the TDS concentration.



**Figure 5.** TDS versus coagulant dose for alum coagulant.

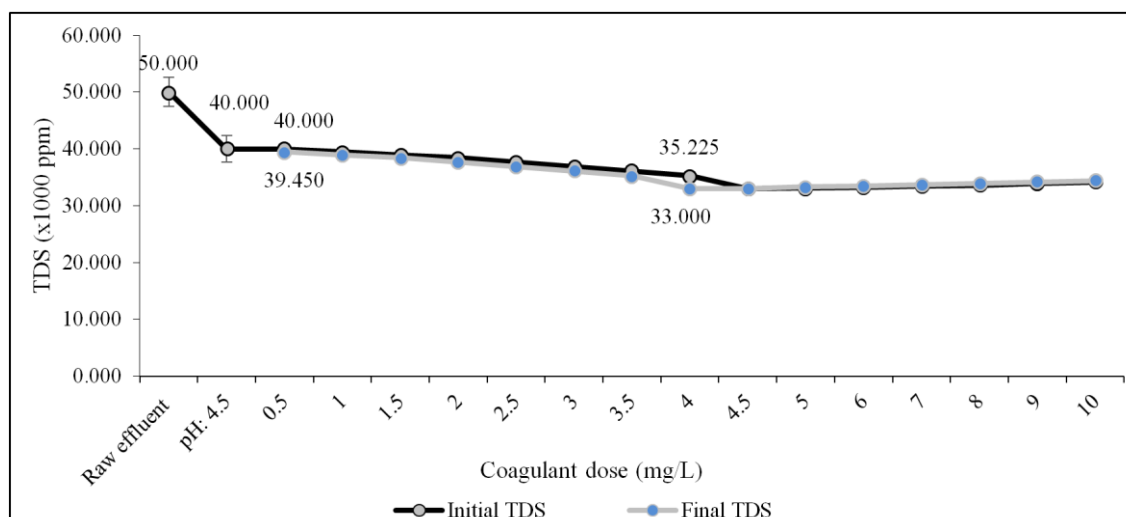


Figure 6. TDS versus coagulant dose for ferric sulfate coagulant.

Figure 6 shows the effect of varying ferric sulfate dose as a coagulant in TDS concentration of effluent. Ferric sulfate is used as a coagulant for various coagulation and flocculation studies [48]. The initial TDS of effluent is 50,000 ppm which reduces to 40500 ppm after pH adjustment to 4.5. The pH value is adjusted to 4.5 as the ferric sulfate coagulant gives maximum TDS removal in the operating pH range from 4.5 to 6 [49]. It is found that the TDS values decrease from 40500 ppm to 33000 ppm with an increase in coagulant dose from 0.5 mg/L to 4 mg/L, respectively. Optimization of coagulant dosage is important to obtain maximum TDS removal efficiency. Coagulant dosage above 4 mg/L will re-suspend settled dissolved and suspended solids into the effluent eventually increasing TDS concentration. TDS reduction efficiency is found to be the same as 34% for both aluminum sulfate and ferric sulfate coagulants, however, the optimum coagulant dose concentration of ferric sulfate (4 mg/L) is less than aluminum sulfate (7 mg/L). A ferric sulfate coagulation process is carried out for the optimi-

zation of various parameters for effective effluent treatment in the current study. Figure 7 represents the variation in pH during the ferric sulfate coagulation process. The studied effluent's initial pH value was 0.10 (Table 1). During the coagulation process with ferric sulfate as a coagulant, a linear relationship between pH and coagulant dose is observed. Ferric sulfate reacts with water molecules present in effluent and forms ferric hydroxide  $\text{Fe}(\text{OH})_3$  [50]. Ferric hydroxide possesses basic characteristics; as a result, the pH value increases with the increase of ferric sulfate coagulant dose. In highly acidic effluent ( $\text{pH} \approx 0.1$ ), metals such as  $\text{Co}^{2+}$ ,  $\text{Li}^+$ ,  $\text{Ni}^{2+}$ , and  $\text{Mn}^{2+}$  from the processing of waste LIBs remain dissolved. Upon pH adjustment and coagulant addition ( $\text{Al}_2(\text{SO}_4)_3$  or  $\text{Fe}_2(\text{SO}_4)_3$ ), these ions precipitate as metal hydroxides (e.g.,  $\text{Co}(\text{OH})_2$ ,  $\text{Fe}(\text{OH})_3$ ,  $\text{Al}(\text{OH})_3$ ). These hydroxides form voluminous, gelatinous flocs with high surface charge, which sweep suspended particles and adsorb dissolved metals through charge neutralization and co-precipitation [51].

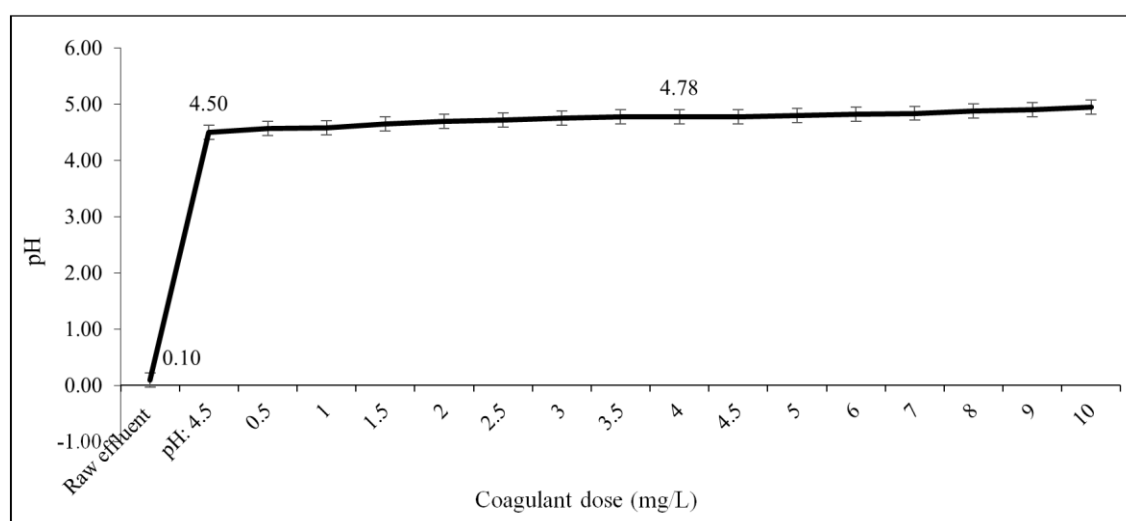
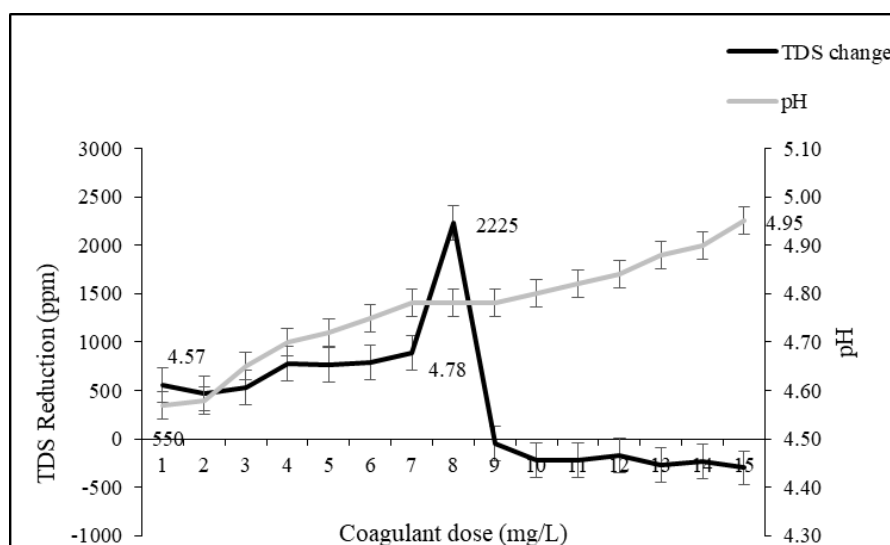


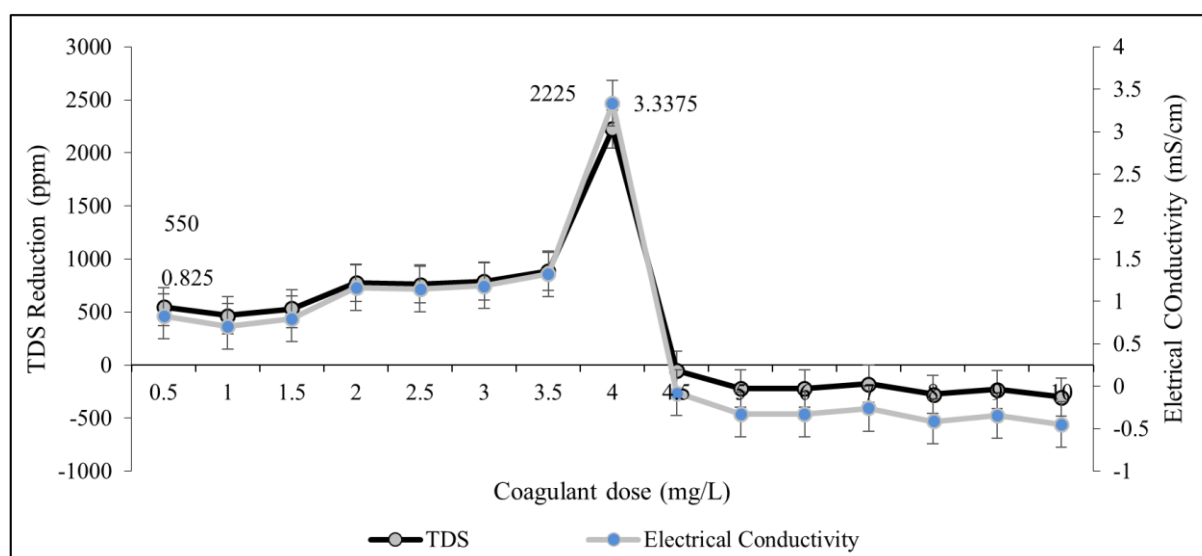
Figure 7. pH versus coagulant dose (mg/L) for ferric sulfate coagulant process.



**Figure 8.** Change in TDS concentration and pH values of effluent with varying coagulant dose in the coagulation process.

Figure 8 shows the variation change in pH and TDS (mg/L) values with varying ferric sulfate coagulant doses ranging from 0.5 mg/L to 10 mg/L. The addition of a ferric sulfate coagulant increases the pH value throughout the coagulation process [52]. The TDS concentration reduction is found maximum at 4 mg/L coagulant dose, which drastically de-

creases with a further increase in coagulant dose to 4.5 mg/L. Negative TDS change concentration represents the final TDS concentration in the effluent is greater than the initial TDS concentration. Resuspension of settled suspended and dissolved solids increases the final TDS concentration of an effluent.



**Figure 9.** TDS and electrical conductivity variation during the coagulation process.

Figure 9 represents a change in TDS (mg/L) concentration and electrical conductivity (mS/cm) during the coagulation process. The graph highlights the overlapping of TDS and electrical conductivity change throughout the coagulation process. Many dissolved ions remain in solution due to electrostatic stability. Coagulants neutralize these charges, causing ions to aggregate and precipitate with other hydroxide flocs. Duan & Gregory reported on coagulation by hydrolys-

ing metal salts and observed that coagulation can remove 15–35% of TDS depending on the effluent composition [53]. In oxalic acid-based effluent (as in the present study), a notable portion of TDS reduction arises from the removal of dissolved cobalt and oxalate ions once pH is increased and precipitation occurs. At higher coagulant dosages, bulk precipitation of metal hydroxide occurs, which can give large flocs of rather open structure. It is found that the TDS concentration and

electrical conductivity of effluent are directly proportional and possess the same behavior with varying coagulant doses [54]. The pH, TDS, and electrical conductivity values as

observed during the coagulation process are tabulated in Table 3. The time required for the coagulation is observed as 21 minutes with subsequent settling in 30 minutes.

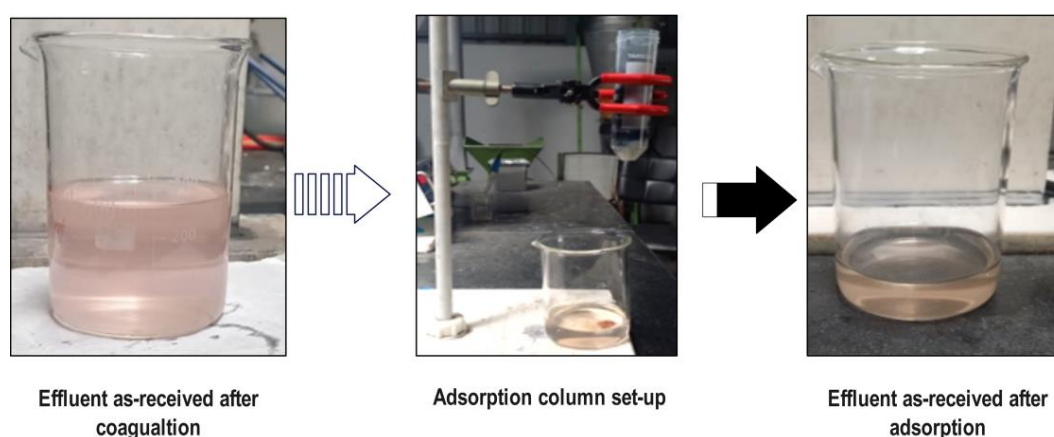
**Table 3.** The pH, TDS, and electrical conductivity values as observed during the coagulation process.

| Sample                                 | pH   | TDS (ppm) | Electrical conductivity (mS/cm) |
|----------------------------------------|------|-----------|---------------------------------|
| Initial effluent                       | 0.10 | 50,000    | 75.00                           |
| Coagulant operating pH                 | 4.50 | 40,500    | 60.00                           |
| As-received effluent after coagulation | 4.78 | 33,000    | 49.50                           |
| Reduction efficiency (%)               | --   | 34        | 34                              |

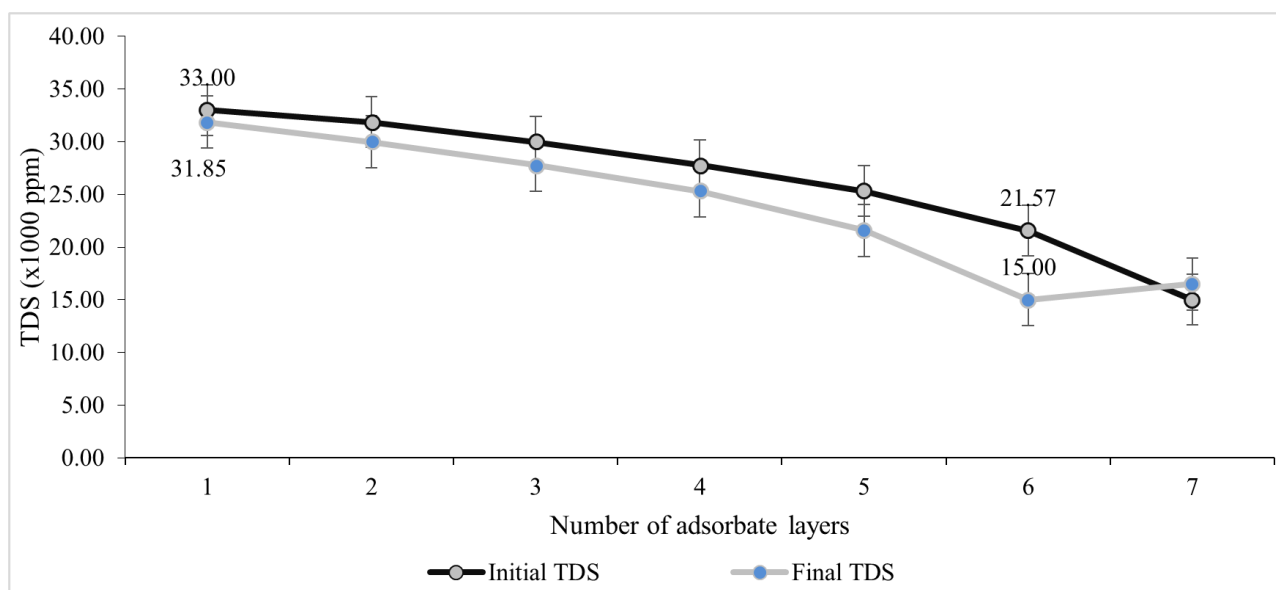
### 3.2. Adsorption Process

The adsorption process acts as a polishing step following coagulation to remove the residual dissolved solids, organic compounds, and trace metal ions from the effluent. In this study, the as-received effluent after coagulation (TDS = 33,000 ppm; conductivity = 49.50 mS/cm) was passed through a column packed with thermally activated carbon derived from waste RO filters. Figure 10 shows the process flow for adsorption treatment of effluent. The TDS removal efficiency from a liquid effluent depends upon the quality and quantity of activated carbon, adsorption column filter depth, TDS concentration, and effluent quantity [55, 56]. Activated carbon possesses a highly porous structure and extensive surface area, which provide numerous active sites for adsorption. Thermal activation at 400 °C removes volatiles and opens blocked pores, thereby increasing both surface area and

pore accessibility, which enhances adsorption capacity. Metal ions (e.g.,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Mn}^{2+}$ ) adsorb through ion exchange and surface complexation with functional groups such as hydroxyl ( $-\text{OH}$ ), carbonyl ( $\text{C}=\text{O}$ ), and carboxyl ( $-\text{COOH}$ ) groups on the carbon surface [57]. Figure 11 represents the initial and the final TDS concentrations with varying stacking layers of the adsorption column used in the adsorption process. The initial TDS concentration of the adsorption process is observed as 33000 ppm which decreases with an increase in stacking layers of adsorbates. An increase in adsorption bed layers increases the efficiency of TDS reduction [58]. The six-layer stacking of adsorbates results in maximum TDS reduction in the adsorption process. The effluent as-received after adsorption treatment was observed with a final TDS concentration of 15000 ppm, which was estimated to be ~55%, TDS reduction from the initial TDS concentration of 33000 ppm efficiency is obtained during the adsorption process.



**Figure 10.** Process flow diagram for adsorption treatment of effluent.

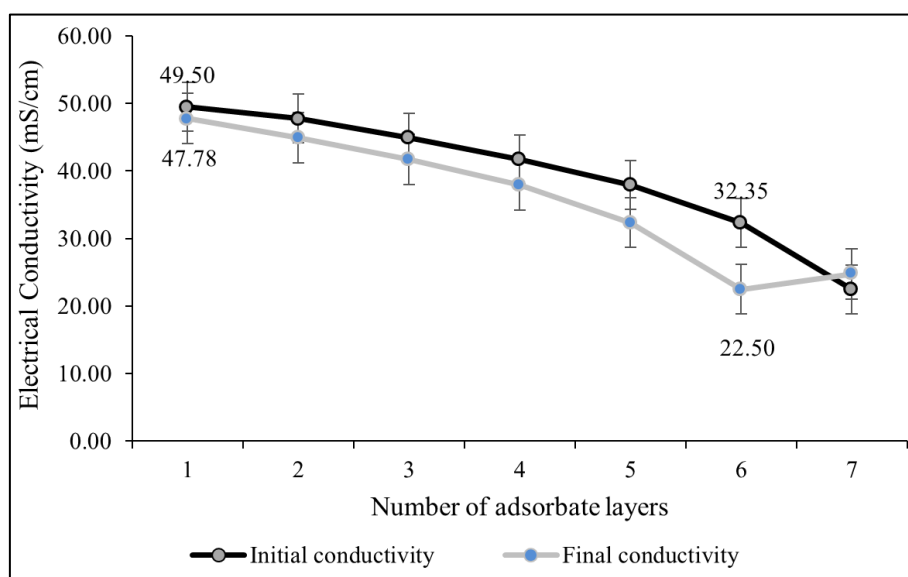


**Figure 11.** Effect of varying adsorption column stacking layers of adsorbates on TDS concentrations in the adsorption process.

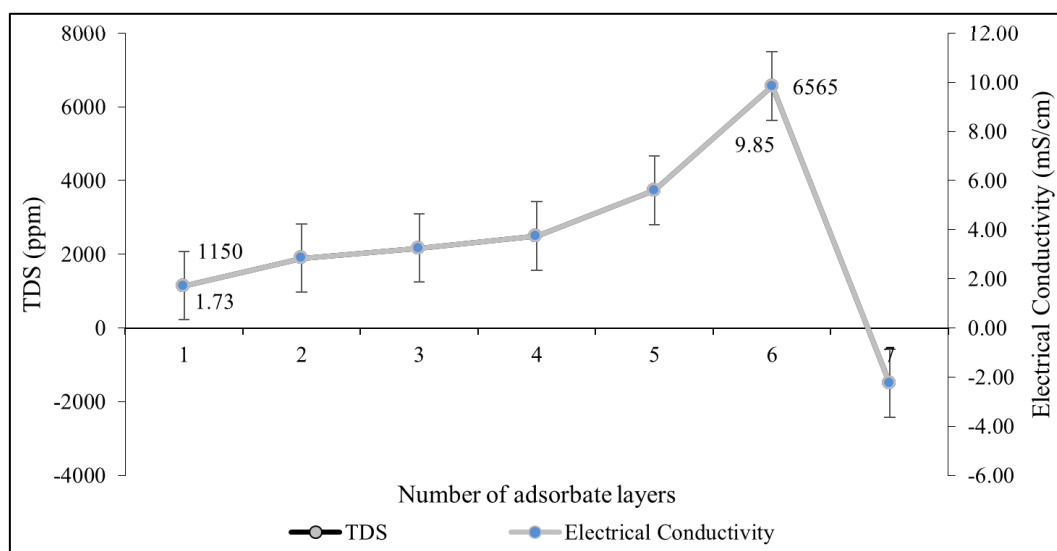
Figure 12 shows the electrical conductivity values of the effluent samples observed during the adsorption process. An increase in the number of adsorbent layers significantly improved TDS removal efficiency. Multi-layer stacking provides longer residence time and enhanced contact between effluent and adsorbent surface. The six-layer configuration achieved the highest reduction, lowering TDS from 33,000 ppm to 15,000 ppm and conductivity from 47.78 mS/cm to 22.5 mS/cm (~53% reduction). Similar effects of increased bed depth on pollutant removal have been reported by Babel & Kurniawan (2003), Mohan & Pittman (2007) and Patel

(2019) [58-60].

Figure 13 represents a change in TDS (mg/L) concentration and electrical conductivity (mS/cm) values of effluent during the adsorption process. The change in TDS and electrical conductivity during the adsorption process follows the same trend. It emphasizes that TDS and electrical conductivity of effluent are directly proportional to each other [58]. The proportional decrease in electrical conductivity indicates that ionic species are the dominant contributors to TDS in the studied effluent. Table 4 illustrates the TDS and electrical conductivity values observed during the adsorption process.



**Figure 12.** Effect of varying adsorption column stacking layers of adsorbates on the electrical conductivity of effluent during the adsorption process.



**Figure 13.** TDS and Electrical conductivity variation during the adsorption process.

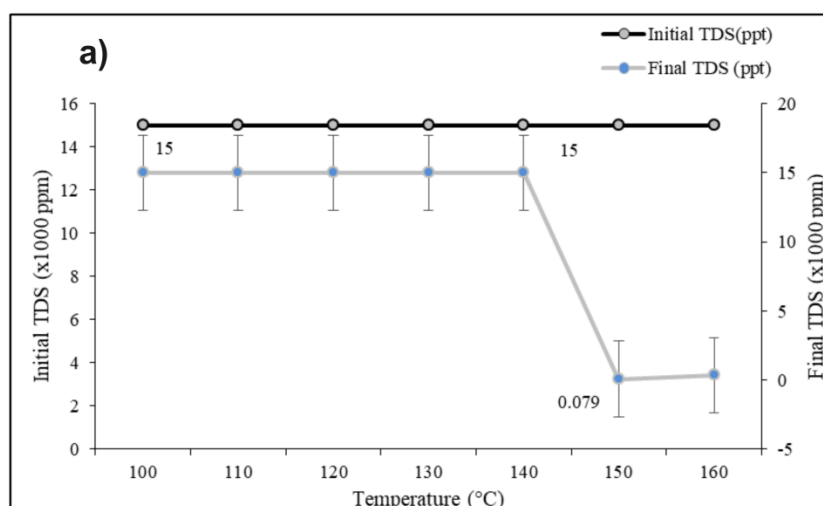
**Table 4.** TDS and electrical conductivity values observed during the adsorption process.

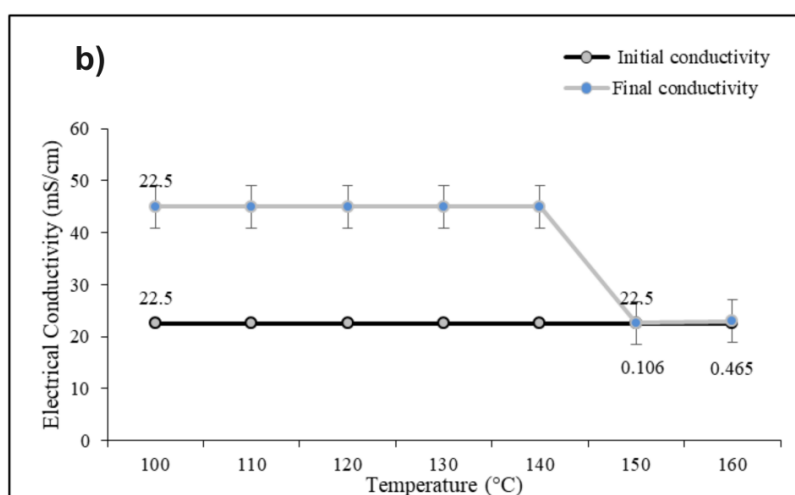
| Sample           | TDS (ppm) | Electrical Conductivity (mS/cm) | Number of adsorbate layers in the adsorption column |
|------------------|-----------|---------------------------------|-----------------------------------------------------|
| Initial effluent | 33,000    | 47.775                          | 6                                                   |
| Final effluent   | 15,000    | 22.500                          |                                                     |

### 3.3. Distillation Process

The distillation step serves as the final and most advanced purification stage in the treatment of oxalic acid-based effluent generated from LIB recycling. After coagulation and adsorption, the effluent still contained residual dissolved salts (TDS  $\approx$  15,000 ppm) and moderate conductivity (22.5 mS/cm). Distillation was employed to achieve complete sep-

aration of non-volatile impurities, metal ions, and dissolved organic residues. Distillation relies on phase separation by vaporization and condensation. When the effluent is heated to its boiling point, only volatile components transition to vapor, leaving behind non-volatile dissolved solids (metal salts, oxalates, and other heavy organic species) in the residue. The condensed vapor produces a clear, low-TDS distillate, while metals and oxalates remain in the bottom concentrate.





**Figure 14.** a) TDS concentration and b) electrical conductivity values with an increase in temperature from 100 to 160 °C during the distillation process.

Figure 14a shows variation in initial and final TDS values with an increase in temperature from 100 to 160 °C during the distillation process. Experimental data showed a drastic reduction in TDS from 15,000 ppm to 79 ppm at 150 °C, corresponding to a >99% removal efficiency. Distillation below 150 °C was ineffective due to incomplete vaporization caused by the high ionic strength and boiling point elevation of the effluent. However, at 160 °C, a slight increase in TDS (to 349 ppm) was observed, likely caused by entrainment or volatilization of certain impurities at elevated temperatures. This could also be due to the dissolution of certain impurities at high temperatures which are transferred to the distilled solution, thereby increasing the TDS concentration [60]. Thus, 150 °C was identified as the optimal operational temperature for maximum purification efficiency.

The electrical conductivity values were also analyzed during

the distillation process as shown in Figure 14b. The initial electrical conductivity concentration of the effluent distillation process is observed as 22.500 mS/cm. The electrical conductivity is found to decrease drastically from 22.500 mS/cm to 0.106 mS/cm, which coincide with TDS behavior, confirming near-total removal of ionic species [30]. Table 5 illustrates the TDS and electrical conductivity values observed during the distillation process. It is found that the final distillate exhibited TDS = 79 ppm and conductivity = 0.106 mS/cm, both well below the CPCB discharge limit of 2100 mg/L. The COD decreased from 1640 mg/L to 32 mg/L, yielding a ~99% reduction efficiency, demonstrating the process's ability to remove both ionic and organic contaminants effectively. The resulting condensate is colorless, low in dissolved solids, and reusable, while the residue can be safely processed for recovery of metal values or solid waste management.

**Table 5.** TDS and electrical conductivity values obtained after the distillation process.

| Sample           | TDS (ppm) | Electrical conductivity (mS/cm) | Temperature ( °C) |
|------------------|-----------|---------------------------------|-------------------|
| Initial effluent | 15,000    | 22.500                          | 150               |
| Final effluent   | 79        | 0.106                           |                   |

**Table 6.** Proposed effluent treatment process results.

| Unit processes | TDS concentration (ppm) |       | Electrical conductivity (mS/cm) |       | pH value (As received effluent pH = 0.1) |
|----------------|-------------------------|-------|---------------------------------|-------|------------------------------------------|
|                | Initial                 | Final | Initial                         | Final |                                          |
| Coagulation    | 50000                   | 33000 | 75.00                           | 47.50 | 6.5                                      |
| Adsorption     | 33000                   | 15000 | 47.50                           | 22.50 | 6.0                                      |
| Distillation   | 15000                   | 79    | 22.50                           | 0.11  | 4.5                                      |

Table 6 summarizes the results obtained for TDS concentration, electrical conductivity and pH variation during the effective effluent treatment process comprising coagulation, adsorption followed by distillation. The distillation process represents the polishing step in the multi-stage effluent treatment, achieving near-complete removal of dissolved and non-volatile contaminants. The optimized operation at 150 °C yields ultra-low TDS and COD values, demonstrating that distillation is an effective method for achieving regulatory compliance and potential water reuse in LIB recycling effluent management. Table 7 shows the ICP-OES results for elemental concentration of various elements present in the untreated effluent as-received after LIB recycling (before treatment) and the effluent as received after treatment (distillation). The ICP-OES results reveal a clear evidence of the substantial reduction in elemental concentrations following

the integrated treatment process. The untreated effluent exhibited high levels of Co, Mn, Cr, Na, Ca, and P, reflecting the presence of residual cobalt oxalate and associated metal salts. After sequential coagulation, adsorption, and distillation, all transition metals—including Co, Mn, Ni, and Cr—were below detection limits (ND) in the distillate, confirming their complete immobilization in the residue. Only trace quantities of Na (22.7 mg L<sup>-1</sup>), Ca (5.0 mg L<sup>-1</sup>), K (5.2 mg L<sup>-1</sup>), and Cu (1.2 mg L<sup>-1</sup>) were detected, likely due to minor entrainment or volatility of soluble salts. This corresponds with the significant decrease in TDS (from 50,000 mg L<sup>-1</sup> to 79 mg L<sup>-1</sup>), demonstrating that the integrated process effectively eliminates both heavy and alkali metals. The results validate the proposed method as a highly efficient metal decontamination and water recovery strategy for acidic, high-TDS effluents from Li-ion battery recycling.

**Table 7.** Elemental concentration analysis of untreated and final stage (distillation) treated effluent.

| Element                      | Results in mg L <sup>-1</sup> |    |      |       |    |    |    |      |      |      |    |      |    |    |    |
|------------------------------|-------------------------------|----|------|-------|----|----|----|------|------|------|----|------|----|----|----|
|                              | Fe                            | Co | Ba   | Cr    | Mn | Ni | Al | Na   | Mg   | Ca   | P  | B    | Cd | Pb | Ag |
| Before treatment             | ND                            | 50 | 0.15 | 10    | 80 | ND | 30 | 100  | 10   | 20   | 80 | 0.07 | ND | ND | ND |
| After treatment (distillate) | ND                            | ND | ND   | 0.123 | ND | ND | ND | 22.7 | 0.05 | 5.01 | ND | ND   | ND | ND | ND |

| Element                      | Results in mg L <sup>-1</sup> |    |    |    |     |    |       |       |       |      |    |    |      |       |       |
|------------------------------|-------------------------------|----|----|----|-----|----|-------|-------|-------|------|----|----|------|-------|-------|
|                              | Bi                            | Cu | Ga | In | K   | Li | Sr    | Tl    | Zn    | Si   | Sn | As | Se   | Sb    | Bi    |
| Before treatment             | 2.126                         | ND | ND | ND | 10  | 20 | 0.006 | 0.076 | 0.017 | 0.12 | ND | ND | 0.04 | 0.124 | 2.126 |
| After treatment (distillate) | 1.21                          | ND | ND | ND | 5.2 | ND | ND    | ND    | ND    | ND   | ND | ND | ND   | 0.1   | 1.21  |

\*ND stands for “not detected”.

### 3.4. Preliminary Technical and Economic Feasibility Analysis

The integrated effluent treatment system comprising coagulation, adsorption, and distillation demonstrated strong technical feasibility at the laboratory-to-pilot scale for treating oxalic acid-based process effluent generated during cobalt oxalate precipitation in LIB recycling. The combined process achieved sequential removal efficiencies of 35–40% TDS and 40–50% COD in coagulation, ~55% TDS and 53% conductivity reduction in adsorption, and >99% TDS and COD removal through optimized distillation at 150 °C, producing condensate meeting CPCB discharge standards. The tech-

nologies employed are modular, reliable, and readily scalable, with an estimated capital cost of ₹30–40 lakhs (USD 30–40K) for a 100 L batch unit and operating costs of ₹25–35 (USD 0.3–0.5) per liter, primarily due to distillation energy demand (~1.5 kWh L<sup>-1</sup>). Despite higher thermal costs, overall economics remain favorable owing to reusability of the distillate, and the use of waste-derived activated carbon, which reduces adsorbent cost by up to 70%. The process cost may increase further with rising electricity tariffs and maintenance of heating systems. However, these limitations can be mitigated in future pilot-scale or industrial applications through several optimization strategies such as incorporating waste-heat recovery systems, multi-effect distillation (MED) or vacuum-assisted evaporation to lower boiling point requirements,

and integration with renewable energy sources (e.g., solar thermal or biomass-based heating). Thus, while energy consumption remains the dominant cost component, the overall process remains technically robust, scalable, and amenable to optimization for future deployment in LIB recycling or similar high-TDS effluent treatment systems.

## 4. Conclusions

The study demonstrates an integrated coagulation–adsorption–distillation process for the effective treatment of oxalic acid–based effluent generated during cobalt oxalate precipitation in Li-ion battery recycling. In the first step, coagulation using  $\text{Fe}_2(\text{SO}_4)_3$  at optimized pH  $\approx$  4–5 achieved 34–40% reduction in TDS and  $\sim$ 45% COD removal through precipitation of metal hydroxides and oxalates. Subsequent adsorption using thermally activated RO derived carbon ( $< 5 \mu\text{m}$ , activated at  $400^\circ\text{C}$  for 1 h) further decreased TDS from 33,000 to 15,000  $\text{mg L}^{-1}$  and conductivity from 47.8 to 22.5  $\text{mS cm}^{-1}$ , corresponding to  $\sim$ 55% and 53% removal efficiencies, respectively. The final distillation step operated at  $150^\circ\text{C}$  produced a colorless condensate with TDS = 79  $\text{mg L}^{-1}$  and COD = 32  $\text{mg L}^{-1}$ . The sequential process effectively reduced pollutant concentrations achieving up to 99% removal of TDS and COD, and producing a final condensate that meets CPCB discharge standards. Preliminary techno-economic assessment confirmed the process to be technically feasible and scalable, with energy consumption in the distillation step identified as the primary cost factor. Future work should focus on pilot-scale validation, integration of heat recovery systems, and process intensification to enhance energy efficiency and overall sustainability of the recycling workflow.

## Abbreviations

|      |                                    |
|------|------------------------------------|
| LIB  | Li-Ion Battery                     |
| TDS  | Total Dissolved Solids             |
| BOD  | Biochemical Oxygen Demand          |
| COD  | Chemical Oxygen Demand             |
| EV   | Electric Vehicles                  |
| TSS  | Total Suspended Solids             |
| CPCB | Centre Pollution Control Board     |
| APHA | American Public Health Association |
| MEE  | Multi-Effect Evaporation           |
| VOC  | Volatile Organic Compound          |
| NF   | Nano-filtration                    |
| RO   | Reverse Osmosis                    |
| CPCB | Centre Pollution Control Board     |
| ND   | Not Detected                       |

## Author Contributions

**Dhvani Purohit:** Conceptualization, Data curation, For-

mal Analysis, Investigation, Methodology, Resources, Writing – original draft

**Kadari Ramaswamy:** Conceptualization, Data curation, Formal Analysis, Investigation, Methodology, Resources, Writing – original draft

**Anoop Satheesh Kumar:** Data curation, Formal Analysis, Investigation, Methodology, Resources, Visualization, Writing – original draft

**Priyadarshini Bais:** Data curation, Formal Analysis, Investigation, Methodology, Resources, Visualization, Writing – original draft

**Ratheesh Ravendran:** Project administration, Supervision, Validation, Writing – review & editing

**Ajay Kaushal:** Conceptualization, Data curation, Formal Analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Supervision, Validation, Visualization, Writing – review & editing

## Ethics Approval and Consent to Participate

The authors affirm that the current study is an analytical study on treatment of effluent generated from processing of waste batteries at lab scale. The study does not include any human participants or biological sample analysis. It is confirmed that no ethical approval is required.

## Consent for Publication

The authors affirm that the study does not include any human participants, their data or biological materials. Authors hereby provide consent for the publication of the manuscript detailed above.

## Availability of Data and Material

The authors confirm that the data supporting the findings of this study are available within the article.

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## Conflicts of Interest

The authors declare no conflicts of interest.

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