

Research Paper

Pre-Treatment and Characterization of Cathode Active Material from Spent Lithium-IoN Batteries

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Abstract— The development of lithium-ion batteries (LIBs) for use in electric vehicles, portable electronic devices, and energy-storage devices has resulted in an increase in the quantity of used LIBs. The environment and public health are seriously endangered by the hazardous metals found in spent LIBs, for example Cobalt and Manganese metals, with a poisonous caustic electrolyte, and binders of organic origin. Based on these factors, procedures for recycling LIBs that are efficient, affordable, also benign to the environment are being developed in order to ensure a long-term sustainable future solution. In this research, used lithium batteries, were released into a sodium chloride solution with a lower concentration, manual dismantling of LIBs components, separations of components as cathode, anode, plastic casing, separators, electrolyte. Also dissolution of cathode material in sodium hydroxide solutions and finally characterized the cathode electrode, using X-ray fluorescence spectrometer (XRF) and Thermogravimetric analysis (TGA). The Polyvinylidene fluoride (PVDF) and diethyl carbonate (DEC) particles in raw cathode material, detected by Fourier Transform infrared (FT-IR) analysis, were effectively thermally decomposed at 464.98 °C and 150 °C respectively. The XRF analysis of thermally treated cathode material powder showed presence of: 61.72 % of Co, 12.11 % of Ni, 9.64 % of Mn, and 16.53 % others component.

Keywords— Lithium-ion batteries (LIBs); Cathode active material (CAM); Polyvinylidene fluoride (PVDF); Diethyl carbonate (DEC); Polytetrafluoroethylene (PTFE)

1. Introduction

In portable devices and electric cars, lithium batteries have become more common [1], [2], [3], [4], [5], due to their benefits in environmentally friendly operations, high energy density, extended storage life [6] compact volume, light weight, vast range of application temperatures [7], and low self- release of power efficiency [8], [9], [10], [11].

According to Chen, Guo 2018, Anode, cathode, separators, and electrolyte are the four main components of lithium batteries. A copper foil anode with a carbon graphite coating and an aluminium foil cathode with a lithium cobalt oxide coating are used as the electrodes. Polyvinylidene fluoride (PVDF), serves as a bond between the cathode material and charge collector [3].

The typical composition of used lithium batteries is 5 –28 % cobalt (Co), 5 –10 % nickel (Ni), 5 –7 % lithium (Li), and 5 –10 % other metals (copper) (Cu), [2] [12], [13], aluminium (Al), iron (Fe), about 7 % plastic and 15 % of dissolved and undissolved organic compounds [8], [9] [14], [15] based on

the manufacturers' specifications, which demonstrate their compositions [7], [16],[17]

Heavy metals, organic compounds, and plastics are all included in waste LIBs, which will cause environmental pollution [8], [18], [19], [20]. Hence, recycling of the principal parts making up, used lithium batteries is seen as a good solution to reduce environmental contamination and a new supply of metals [21]. Statistics show that LIBs only have a one- to three-year lifespan in digital items. [2], [8], [9] [19], [20] and a five- to eight-year lifespan in power vehicles. [8], [9], [22].

Toxic heavy metals may enter underground water tables, due to chances of the land-filling of used LIBs [23]. Burning used LIBs will produce a significant number of hazardous gases, including hydrogen fluoride (HF) gas, which will contaminate the atmosphere [23], [24], [25]. Due to the enormous quantity of rich metals found in spent LIBs, where some of which are even of a better grade than those found in natural ores, they have a high economic worth. [8], [9], [22], [26]. Comparatively speaking, the other conventional forms of batteries, LIBs have traditionally been seen as cleaner and

greener energy storage technologies. [22], [27]. However, based on the dangerous elements they contain, LIBs continue to be a concern to both the environment and public health [28]. Therefore, in order to save their primary components, it is vital to safely recycle used LIBs. [8], [9], [26], [27], thus preventing dangerous organic substances from the electrolyte, separator, and binder, as well as the cathode active material that contains heavy metals, from being released into the environment [29] [30],[31] [32][33][34],[35]

2. Related Work

According to shuva A; lithium batteries were mechanically disassembled by removing the plastic covering, using a sharp edge cutting device, by fixing the battery in a vice. After that the different parts were then isolated based on their characteristic appearance [36].

According to X. Chen 2019a; the composition of the collected cathode paste was just determined by using XRD (range of $10^\circ - 90^\circ$, at 2θ using interval of 0.05°) and not XRF, since lithium was present in the compound, which is limited by the XRF analysis [31], [37], [38].

Chabhadiya 2021 and Asadi 2020; reports the use of saturated solutions of sodium sulphate or sodium chloride salts of 10 % wt. and pyrolysis at 300°C , as the methods of neutralizing the charge available in the batteries before subjecting them to any form of treatment [7] [39], [8], [26], [27]. Qiu and San; proposed application of pyrolysis under vacuum, to aid in the weakening of the adhesive force holding cathode material and the charge collector, though a lot of toxic gases like hydrogen fluoride were released [40].

3. Experimental Method

Figure 1, shown below indicates how the sampled used lithium batteries were primarily pretreated in various solutions and finally characterized

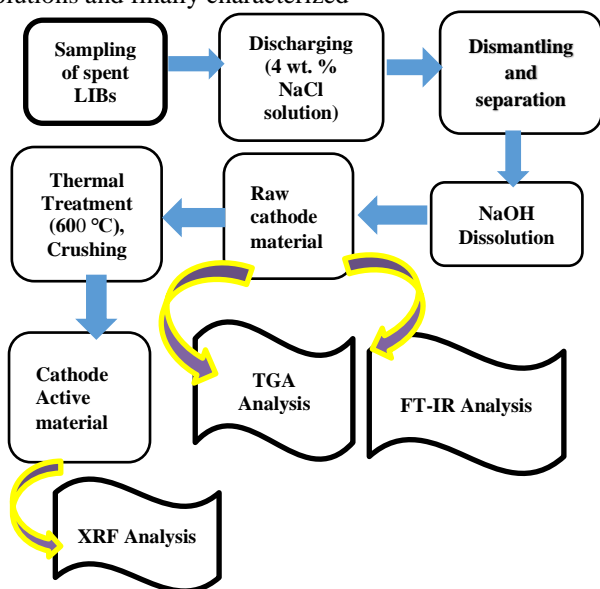


Figure 1: Experimental flow chart for the research design

3.1 Materials and reagents

The already used lithium batteries were collected from mobile phone repairing shops within Nyeri town (Kenya). Sodium chloride for discharging and sodium hydroxide for dissolution of aluminium foil charge collector, in which all of them, were of analytical quality. Salt solutions were made with specific concentrations using deionized water.

3.2 Pre-treatment process:

Discharging of LIBs in Sodium chloride solution.

A total of sixty-seven used lithium batteries were used, where the initial voltage V_o of each was determined before discharging in which spent LIBs samples weighing 1372.85g were all immersed in sodium chloride solution [39] of 4 % wt. for 36 hour [7], [8],[9], [40], [41]. After which the final voltage V_t was determined after the process of neutralization. The residual voltage percent (E_r) and percentage of voltage drop (E_r) of each spent LIBs were determined.

3.3 Manual dismantling of LIBs

The components of used LIBs were disassembled manually, [42] and the respective parts isolated depending on their colors. The cathode, anode, and other parts were disassembled into the following groups: [7]: First, the battery's plastic covering was stripped off. A lathe was then linked to the battery. Using a saw, the battery's end was cut off, and the outer shell was then removed by opening the battery lengthwise. [9].

Last but not least, the cathode and anode were isolated before being dried in an oven at 60°C for about twenty-four hours. [9], [43]. The produced cathode and anode were isolated further, and grouped on a basis of appearance and information available in the published literature [36]. The binder, polyvinylidene fluoride or polytetrafluoroethylene, typically attaches the cathode material to aluminium foil, contributing to challenging removal of the cathode material from the collector [1], [8], [9]. Application of the sodium hydroxide dissolution process allowed the cathode material to be adequately isolated from the foil.

3.4 Sodium hydroxide dissolution of cathode material

The obtained cathode material attached to aluminium foil with aid of binder, were then immersed in prepared 10 % wt. NaOH solution for 30 hours, to aid in the dissolution of aluminium foil from the material thus weakening the binder [22], [33], [35].

3.5 Thermal analysis of cathode material

Thermogravimetric analysis (TGA), was performed in a TGA - 50 series (SHIMADZU) equipment, using samples of 26.228 mg and under nitrogen atmosphere supplied at a 50 mL per minute. Samples were subjected to heating between 22.87 up to 600°C , with an aim of preventing the fragile nature of undissolved aluminium traces, which might lead to complications for the further steps.

3.6 Characterization of the cathode material

An X-ray fluorescence spectrometer (XRF BRUKER 00466 - Geochem. PdZ), was used to analyze the elemental contents of the used lithium batteries cathode materials, for both raw

and thermally treated cathode material. This was conducted by placing 5g of the powder sample into the glass disks and mounting onto the holder for analysis. The presence of PVDF and diethyl carbonate (DEC), were analyzed using FT-IR (PerkinElmer Co.), by placing 0.1g of the dry prepared spent LIBs powder onto the ATR sample holder for analysis and taking the readings. Thermal stability and degradation lifetime of PVDF and DEC, was evaluated using TGA, as explained in section 2.5 above.

4. Results and Discussions

Effect of discharging spent LIBs in 4% wt. NaCl

Table 1 below shows the residual voltage percent (E_t) and percentage of voltage drop (E_r), obtained during discharging of used LIBs in 4 % wt. sodium chloride solution.

Table 1: The residual voltage percent (E_t) and percentage of voltage drop (E_r), during discharging of LIBs in 4 % wt. NaCl

LIBS No:	V_o (v)	V_t (v)	E_t	E_r
1	2.963	0.211	7.121	92.879
2	3.204	0.245	7.647	92.353
3	3.614	0.244	6.752	93.248
4	2.314	0.276	11.927	88.073
5	1.983	0.261	13.162	86.838
6	2.002	0.256	12.787	87.213
7	3.326	0.266	7.998	92.002
8	3.142	0.312	9.930	90.070
9	2.791	0.286	10.247	89.753
10	2.011	0.297	14.769	85.231
11	2.276	0.291	12.786	87.214
12	1.558	0.243	15.597	84.403
13	1.089	0.245	22.498	77.502
14	3.046	0.283	9.291	90.709
15	2.946	0.394	13.374	86.626
16	3.117	0.401	12.865	87.135
17	2.773	0.298	10.746	89.254
18	3.132	0.385	12.292	87.708
19	2.428	0.319	13.138	86.862
20	2.883	0.271	9.400	90.600
21	1.000	0.220	22.000	78.000
22	1.201	0.216	17.985	82.015
23	0.012	0.001	6.667	93.333
24	0.067	0.008	11.791	88.209
25	0.039	0.001	2.308	97.692
26	0.974	0.102	10.472	89.528
27	2.715	0.243	8.950	91.050
28	1.732	0.209	12.067	87.933
29	1.101	0.212	19.255	80.745

30	2.151	0.304	14.133	85.867
31	1.095	0.231	21.096	78.904
32	0.413	0.096	23.245	76.755
33	2.001	0.246	12.294	87.706
34	1.980	0.276	13.939	86.061
35	2.996	0.302	10.080	89.920
36	2.471	0.293	11.858	88.142
37	3.412	0.341	9.994	90.006

Through comparing the voltage left [13],[46] over after the discharge to the battery's initial voltage, the residual voltage percentage (E_t) in the battery was computed [13], [41]. The residual voltage percentage was determined using the formula below [13]:

$$E_t = \frac{V_t}{V_o} \times 100 \% \quad (1)$$

where V_o represents the LIB's starting voltage and V_t represents the final voltage. The following calculation formula was used to compute the percentage of voltage loss (E_r) as well [13], [41]:

$$E_r = 1 - E_t = 1 - \left[\frac{V_t}{V_o} * 100 \% \right] \quad (2)$$

The percentage voltage drop range was between 93.24 % to 76.76 % while average voltage drop was 87.50 %. According to Table 1, we focused on the use of lower concentrations of 4 % wt. NaCl for 36 hours, to aid in minimizing the high effect of corrosion of the battery electrodes, leakage and pollution. This reduced reactivity during the discharging process, thus no traces of electrode corrosion and leakage chances, was detected [13], [41]. This was according to Asadi Dalini 2020, Zheng 2018 and Yuliusman 2018, while using 7–10 % wt. of NaCl for 24–36 hours [44]. Even though the use of lower concentration of NaCl solution takes more time to neutralize the batteries, the batteries were successfully discharged as compare to the use of sodium sulfide or magnesium sulfate according to Torabian 2021. This is due to the fact that, the number of free mobile ions present in the solution of sodium sulfide and magnesium sulphate solution, are comparatively a bit lower compared to the number of ions in the sodium chloride solution. The higher number of free mobile ions in NaCl solution, results in a more enhanced electrons transfer, attributing to a higher and stronger neutralization of used lithium batteries [17], [44].

Cathode and anode terminals, have higher chances of short-circuiting if the batteries are not discharged, [13], [41] which would unleash chemical energy that has been stored and result in a fast temperature increase, gas release, fires, or even potentially devastating explosions, [2], [45], [17], [29], [34], [9]

4.2 Manual dismantling of LIBs

Figure 2, below shows the various components of disassembled used lithium batteries, indicating the anode, cathode and the batteries casing.

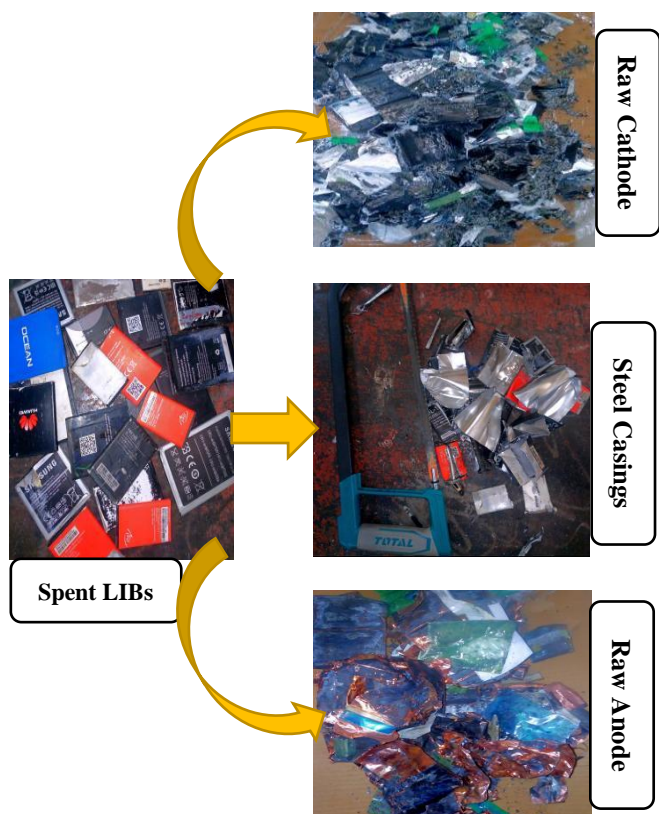
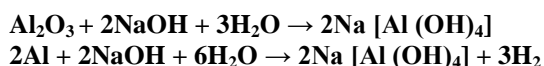


Figure 2: Manual dismantling of LIBs and separation of anode, cathode and steel-casing

The discharged lithium-ion batteries were dismantled as shown in figure 2, while separating each component according to the colour of each charge collector, for example anode has brown - copper as charge collector while cathode has a white - aluminium foil.

4.3 Effect of Sodium Hydroxide dissolution cathode material.

The 10 % wt. sodium hydroxide dissolution was carried out to dissolve the aluminium foil and thus weakening the binder, hence exposing the cathode material, with aluminium dissolving as per the equations below:



Aluminium's amphoteric characteristic makes sodium hydroxide effective at separating the materials. [8], [9], [22], [26]. The leftover cathode material that had not yet been dissolved was dried in an oven for 24 hours at 60 °C.

4.4 FT-IR analysis of raw cathode material

Figure 3, below shows the FT-IR spectrum for the determination of the Polyvinylidene fluoride, and diethyl

carbonate present in the raw cathode material before subjecting to thermogravimetric analysis.

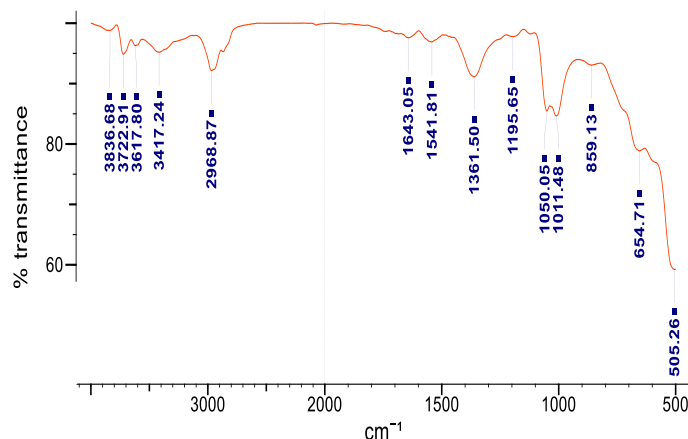
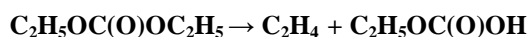


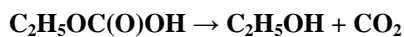
Figure 3: FT-IR Spectrum of Raw Cathode Material

The FT-IR (PerkinElmer Co.) spectra with a resolution of 4 per-centimeter and operating between 4000 and 500 per-centimeter for raw cathode material, was used to evaluate the presence of PVDF and Diethyl carbonate (DEC).

The OH bond of ethanol, from DEC disintegration, were at 3837.65 cm^{-1} (**O-H** sharp) and 3721.94 cm^{-1} (**O-H** broad),[47] while the absorption of carboxylic acid group's **O-H** bond is located at 3626.48 cm^{-1} [48]. The -C-H bond of alkane and broad **O-H**, with **C-H** stretches, of carboxylic acids gives absorption at about 2968.87 cm^{-1} and 2869.56 cm^{-1} respectively. The absorbance band at 1644.02 cm^{-1} shown above, is as result of carboxylic acids and esters carbonyl groups (**C=O**), [49]present in the raw cathode material. These, were closely similar in relation to what, Lie & Liu, 2021, findings of the major characteristic regions as; in the esters of DEC, the stretching vibrations of alcohol O-H are shown by the range 3672-3372 cm^{-1} , C=O by the range 1726-1620 cm^{-1} , and C-O by the range 1419-1400 cm^{-1} . [12]. Ethene with C=C stretching absorbed at 1551.45 cm^{-1} of wavelength in the lower frequency. In fingerprint region (400–1400 cm^{-1}), the C-C stretches, C-H bending vibrations, absorption occurred. For example, in PVDF, the CH₂ bend at 1390.42 cm^{-1} , C-F out of plane at 653.75 cm^{-1} , while in ethene, the absorption bands at 1015.34 cm^{-1} for =C-H. At 1196.61 cm^{-1} is the absorption band for C-O stretch of carboxylic acid at 1051.98 cm^{-1} for C-O in alcohol and ester. Diethyl carbonate is an ester of carbonic acid and ethanol, which further disintegrates into ethene, ethanol. Lithium hexafluorophosphate is generally dissolved in alkyl carbonates to form the electrolytes of lithium batteries. [18], [20], [26], [31], [37], [38], [50], [51] Since the carbonates are combustible, thermal runaway is a possibility during high-load operations like quick charging or overcharging. DEC primarily decomposes into C₂H₄ and the alkyloxy group as follows:

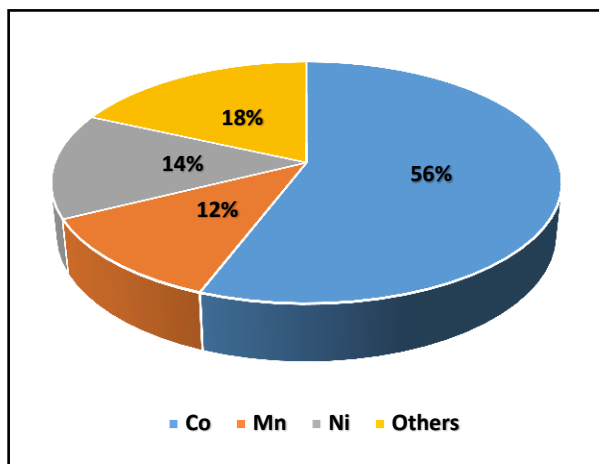


When this happens, the alkyloxy group quickly breaks down into C₂H₅OH and CO₂:

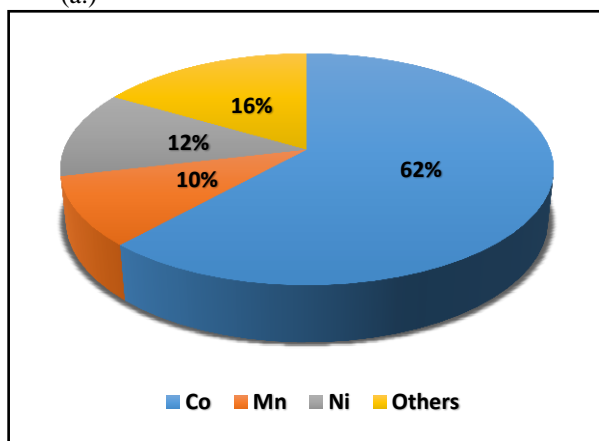


4.5 XRF Analysis of raw and thermally treated cathode powder

Fig 3 shows the metal composition of the raw and thermally treated cathode material of spent LIBs. XRF analysis of the cathode revealed presence of Co, Ni, aluminium oxide and Mn. This is because doping and surface modification were employed to boost the lithium batteries capacity.



(a.)



(b.)

Figure 4: XRF Analysis of: (a.) Raw Cathode Material and (b.) thermally treated cathode material.

In used lithium batteries cathode material analyzed, cobalt metal made up the majority of the metals (61.72 %), with 12.11 % nickel, coming in second. 9.64 % manganese coming third, and finally 16.53 % others component, as shown in Fig 4 above for XRF analysis. However, the atomic number of the element's limits XRF, therefore substances like Li, carbon, and oxygen that have low atomic numbers and low energies cannot be detected by XRF. [12], [52], [53], [54]. The higher concentration of heavy metals in these used lithium batteries, calls for a more attention of developing an environmentally friendly recycling methods of the huge masses of used batteries to minimize the disposal of contaminants into the ecosystem

These compositions were within the range of what is reported in literature, as captured in Fu 2019a and Chen 2018a

journals, with weight percentage composition at 59.55 wt.% Co, 0.32 wt.% Mn and 1.21 wt.% Ni after calcination, and also (Lie & Liu, 2021) obtaining (62.65 %) of Co. The breakdown of lithium salts in the electrolyte and the vaporization of organic solvents, separator material, polyvinylidene fluoride (PVDF), all occurs during thermal treatment process, hence, the variation in elemental composition before and after calcination.

4.6 TGA analysis of raw cathode material.

Figure 5, below shows TGA thermogram for the thermal decomposition of raw cathode material, with a plot of percentage weight loss as a function of temperature [55].

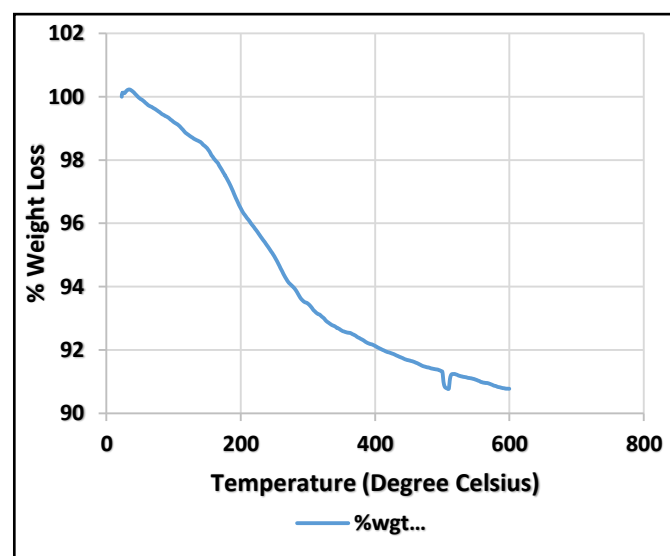


Figure 5: TGA thermogram for thermal decomposition of raw cathode material

The analysis was to evaluate thermal stability and degradation lifetime of PVDF and DEC particles in raw cathode material. (Sattar et al., 2019), [19], [26], [31], [32], [38], [50]. The thermal loss of the esters component in the electrolyte causes the first weight loss stage, which is characterized by temperatures between 53 °C and 150 °C. [31], and trapped water, leading to the process of dehydration and in general a 17 % weight loss.

The TGA curve shows two more other weight losses as the temperature rises. The second weight loss on the curve happens at roughly 315.15 °C, which is in close range temperature at which PVDF thermally decomposes over 300 °C [56], therefore, this suggests that PVDF's heat degradation can be responsible for the second weight loss. The $(\text{CH}_2\text{CF}_2)_n$ of PVDF, disintegrates under heat influences into C-H scission and H-F loss occurring. The final disintegration temperature for PVDF membrane was at 464.98 °C, where the full degradation of the C-F backbone is seen, as shown from the TGA curve [6], [57].

At roughly 520 °C, which corresponds to the third weight loss, the curve clearly shows an exothermic peak. Acetylene black and conductive carbon's combustion-induced breakdown is responsible for the cause [56]. Hence, this

facilitate the removal of bound water, PVDF binders, acetylene black and carbonaceous material from the raw cathode material, thus enhancing further purification of the final cathode active material

4. Conclusion and Future Scope

The results from XRF analysis shows that cathode material has higher composition of highly valuable metals in the order of 61.72 % Co, 12.11 % Ni, 9.64 % Mn, and 16.53 % as other components, hence need of recycling the cathode. This is in order to obtain the most significant or abundant metal(s) and to minimize chances that can enhance a situation whereby, heavy metals can get released into the ecosystem.

Sodium hydroxide dissipation process, successfully facilitated the weakening of the firm binder force holding the charge collector and cathode material. The FT - IR analysis and thermogravimetric analysis, clearly shown the presence of Polyvinylidene fluoride and Diethyl carbonate, this can be successfully eliminated through thermal disintegration as evidenced by TGA analysis.

Conflict of Interest

No any conflict of interest was encountered by the authors.

Acknowledgments

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