

INDIAN INSTITUTE OF TECHNOLOGY GANDHINAGAR

PROJECT REPORT by Group C CL -201

PROCESSES INVOLVED IN MANUFACTURING OF LITHIUM FOR BATTERIES

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Abstract

Lithium-ion batteries (LIBs) have become a modern-day solution for almost every energy storage problem. The applications of LIBs have increased rapidly, and it is expected to follow a similar trend in the upcoming time. Therefore, a sufficient supply of high-quality Lithium (99.5% pure) and other battery components is vital. Currently, there are some predominant sources of Lithium that are generally used in the manufacturing of Lithium. South American countries like Chile and Argentina contain vast reserves of Lithium in the form of lithium brine which constitutes about 43% of the world's total reserves. These countries also have mines that produce ores like Spodumene, Petalite, Lepidolite and several other lithium-rich minerals. Currently, around 5% of the total Lithium is also incurred from recycling the lithium batteries. However, this percentage is bound to grow in the future. Lithium manufacturing is still a very young industry, and therefore, it has a massive scope of development.

Problem Statement

With the increasing demands of lithium-ion batteries, the need to increase the supply of battery-grade lithium is also increasing. However, the capacity of manufacturing more lithium is increasing at the same rate. Therefore, it becomes necessary to understand the current processes used to manufacture lithium to make these processes more efficient and cost-effective with further research. Hence, through this project, we have attempted to understand the current industry standards, the current chemical and physical processes, and the existing limitations of the present procedure. Following are the objectives of this project:

- 1. Studying why graphite, Cobalt and certain other materials are predominantly used in LIBs comparing them with other types of LIBs on the grounds of their specific energy output, storage capacity, cost-effectiveness and safety;
- 2. Studying the entire extraction and segregation process of lithium salts from the ground and making a mass balance chart depicting the entire process;
- 3. Exploring the possibility of recycling LIBs and the process involved in them. Studying the material balance of these processes;
- 4. Analyzing and making the energy balance charts for the internal reactions of LIBs;
- 5. Analyzing the most recent advances made in the field of research of LIBs and studying the prospects of growth based on specific energy outputs, cost-effectiveness and safety.

Table of Objectives

S.No.	Objectives	Completed
1	Studying why graphite, Cobalt and certain other materials are predominantly used in LIBs comparing them with other types of LIBs on the grounds of their specific energy output, storage capacity, cost-effectiveness and safety.	YES
2	Studying the entire extraction and segregation process of lithium salts from the ground and making a mass balance chart depicting the entire process.	YES
3	Exploring the possibility of recycling LIBs and the process involved in them. Studying the material balance of these	YES

	processes.	
4	Analysing and making the energy balance charts for the internal reactions of LIBs.	NO
5	Analysing the most recent advances made in the field of research of LIBs and studying the prospects of growth based on specific energy outputs, cost-effectiveness and safety.	YES
6	Studying the structure, chemical reactions, and processes that occur inside different types of batteries.	х
7	Going over the various energy outputs of batteries to identify their efficiencies and know-how feasible they are.	х
8	Studying various processes used while manufacturing Li-ion batteries.	х
9	Looking out for why the charging capacity of Li-ion batteries reduces with time and understanding the ongoing research works to solve the problem.	х
10	Examining the actual mechanisms and processes used in an EV to produce mechanical output from electrical energy and analysing their conversion efficiency from one form of energy to another.	X

Modifications in objectives

The objectives of the project have been extensively changed. They are mainly changed due to the flaws in the earlier objective statements. The earlier ones were very vague and did not describe the motive. Moreover, they seemed to not align with the contents of the course. Therefore, the modified objectives have been decided while keeping in mind these points. In the new objectives, we have emphasised the processes we cover, such as extraction of lithium, recycling used Lithium-Ion Batteries and analysing the material balances and outcomes of various LIBs. Other than this, we have also tried to cover objectives that are solely for knowledge purposes.

Unfortunately, we have excluded **Objective 4** as well from the final project. This is because the Lithium-based battery industry is very newly established. Therefore, there exist minimal data on the topic. Further, the very limited available data contained information that appeared to be well beyond our scope. Hence, we took a tough call of excluding objective four which primarily focussed on energy balancing of internal reactions of Lithium-ion batteries.

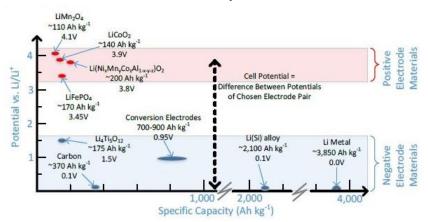
Motivation and Background

The predominant use of certain materials in LIBs Background

We studied how perfectly graphite satisfies all the needs with its unique characteristics. Graphite has some features due to which it is predominantly used in anode of Li-ion batteries:

- 1. Excellent porosity and conductivity- Porosity refers to letting something through it, and here for Lithium-Ion batteries, it relates to the holding capacity of lithium ions. Higher porosity improves the conductivity as it allows a good flow of electrons.
- 2. High durability and lightweight- The material should be light in weight and entirely sustainable. It must stand for a long time without much deterioration and requires minimum maintenance.
- 3. Low cost- Graphite is a sufficiently affordable material.
- 4. Appropriate specific capacity- Graphite's voltage matches (approximately) with that of lithium.
- 5. Active material- To make the battery reversible, we need active material.

Cobalt in cathode material with lithium is an essential element as cobalt oxide (CoO2) in LIBs. Its function in the cathode is very similar to that of graphite in anode, i.e., it easily intercalates lithium in between the layers of CoO2. Also, it provides a perfect balance between positive potential and specific capacity. Therefore, it is hard to substitute Cobalt for any other element.



Unfortunately, Cobalt is a rare element, and its extraction is also an expensive process, thus, making it a costly component to use. Therefore, extensive research is being held in this field to find an appropriate alternative. Some notable mentions of these researches are Lithium Nickel Oxide (LiNiO2) and Lithium Iron Phosphate (LiFePO4). Though, none of these has as good a balance between specific capacity and positive potential. Therefore, cobalt oxide continues to be the most widely used component for the cathode.

Motivation

Some of the research papers have been published in some fields of LIBs which inspire us to do research:

- 1. graphite is predominantly used in the anode.
- 2. The necessity of anode and cathode material for effective LIBs.

- 3. How Cobalt stabilises cation and is used as a cathode material.
- 4. An alternative option for Cobalt in the future is the cathode material.

The extraction process of Lithium from Lithium Brine

Background

Research on the most crucial part of Li-ion batteries- Lithium salt extraction. The extraction of lithium is a very capital and area-wise intensive job. There are various sources of lithium extraction, which are as follows:

- 1. Underground brine reservoirs;
- 2. Minerals and ores from mining like Spodumene, Petalite, Lepidolite;
- 3. Geothermal and oilfield brines.

Of these three sources, underground brine reservoirs are the most significant source of lithium, and they contribute to about 59% of the world's demands. This is because lithium brines contain the highest lithium concentration (roughly between 1000-1500 ppm) among all the sources, thus, making them the most viable lithium source.

South American countries, i.e. Bolivia, Chile and Argentina, are some of the lithium-rich countries in the world. As per the estimates, they contain close to 45 million tonnes (or around 50% of the total world reserves) of lithium reserves. However, not all of it is feasible to extract with the current technology. Because of this, Bolivia, Chile and Argentina have become the extraction sites of lithium for the world.

Today lithium-ion batteries are used in almost every electronic device, e.g., mobiles, laptops, household electronic devices, EVs, and practically any other electronics. This has led to an exceptional increment in the demand for lithium batteries. So, to increase the supplies, there have been several new additions to the existing extraction process to increase the yield of lithium from its brine. One example of the latest inclusions is Reverse Osmosis (RO) to concentrate lithium in brine.

Motivation

Some of the research papers have been published in some fields of LIBs which inspire us to do research:

- 1. Lithium Salt Extraction.
- 2. New technologies that could reduce lithium from brine extraction more economically.
- 3. Novel approaches for Lithium extraction from salt-lake brines.

Recycling processes of LIBs

Background

Research in the recycling of the LIBs and studying all the processes in the recycling of LIBs. Difficulty in recycling lithium-ion batteries:

- The different mixture of materials also complicates battery recycling. Even though all li-ion batteries contain lithium, still other components may vary. In turn, this would also raise the cost of recycling.
- Why is the recycling of Lithium-ion batteries less common?

- Availability of Cheaper Raw Materials: If the cost of new Cobalt were cheaper, recycling
 processors would not compete, and the recycling business would not operate economically.
 Opportunities for the emergence of new chemicals threaten the recycling business. For example,
 if Cobalt is wholly removed from batteries, recycling will not motivate it to remove it from
 battery waste. If other battery chemicals use a different combination of more popular materials
 than lithium-ion, there will be less motivation to replace the materials used in discarded batteries.
- 2. Complex chemistries: Different combinations of materials also make it difficult to recycle the battery. Although all li-ion batteries contain lithium, some components may be different. Batteries consist of various metals such as nickel, cobalt, iron, aluminium, and more. With ever-changing battery chemists, making an efficient extraction process is challenging, as they need to be adapted to each acquired asset. Next, this will increase the cost of recycling and make it less profitable.
- 3. Complicated processes: The structure of the lithium-ion batteries also presents another obstacle before active recycling. The cell components of a battery the cathode, anode, separator, electrolyte are usually rigidly packed together and are not designed to be easily disassembled. There are also different cell designs and configurations. Larger battery packs, such as those in electric cars, can hold thousands of these cells, further complicating the process. Each cell design will require different scattering processes and performance scales, making planning an efficient and effective recycling process.

Recent advancements in the field of research on LIBs Background

With the increasing demand for energy storage, particularly from the rising popularity of electric vehicles, intensified research is required to develop next-generation Li-ion batteries with dramatically improved performances, including improved specific energy, volumetric energy density, cyclability, charging rate, stability, and safety.

Some of the Advancements made in the research field of LIBs:

Conductivity: To enhance the electronic and ionic conductivity of LiFePO4, two common strategies are developed, namely doping by ions and coating by carbon. The electronic conductivity of LiFePO4 can be increased by a factor of 108 by cation doping.

Silicon-based Anode: Recently, much research focus on anode materials has been shifted to silicon-based anode. Silicon has a high theoretical capacity of 4200 mAh/g (lithiated to Li4.4Si) or 3572 mAh/g (based on Li3.75Si), which is about ten times that of graphite and almost four times that of many metal oxides. Protective circuits: It may not be safe if rapidly charged at a low temperature. Therefore, protective circuits are typically used to avoid overcharge and thermal runaway. However, the protective circuits could add weight burdens and decrease the energy density of the whole battery.

Safety: Possible venting and fire when crushed, which requires critical safety enhancement. The recent fires in Li-ion battery packs upon being crushed by metal objects in the promising Tesla Model S cars highlights the importance of battery safety.

Methodology

Extraction of Lithium from Lithium-Brine:

Assumptions:

- 1. The process is approximated to be steady-state because the process is happening for a continuously flowing feed into the inlet. Almost similar processing and reactions will occur for the entire stream entering the system.
- 2. Reactions only occur inside precipitators. This is taken because of the lack of data in the case.
- 3. Boron and sulphate are considered inseparable parts of the residual brine. This is because these compounds' values are minimal compared to other significant components considered in the process.
- 4. Lithium is not released in any intermediate steps/reactions because the lithium lost in the intermediate steps is not very significant compared to other essential components. This reduces all the additional laborious calculations, which would not make much difference to the results.

Process Approximations:

The actual process involved several blocks and streams. We have grouped and merged them into just four blocks. The reason was that the reactions happening was out of our reach of knowledge. However, we combined those reactions with other blocks to reach approximately the correct answers.

Processes involved in Recycling of Lithium Batteries:

Assumptions:

- 1. The process is approximated to be steady-state because the process is happening for a continuously flowing feed into the inlet. Almost similar processing and reactions will occur for the entire stream entering the system.
- 2. The percentage written for Li, Co, Cu in all streams concerns the initial mole flowrate entering as fresh feed.
- 3. The composition of Li, Cu, Co in streams A2, D11 & 13E is assumed to be zero because of lack of data.

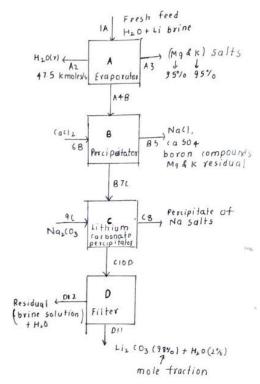
Process Approximations:

The actual process involved several blocks and streams. Nevertheless, we grouped and merged them into just five blocks. We were not familiar with the details of all the reactions present in the recycling of lithium batteries. Some examples of such reactions are the reaction of cobalt with cyanic; the use of D2EHPA to remove Iron and Aluminium. Moreover, the limited availability of data of several elements significant constraint.

Following are the calculations for material balancing of the processes:

- 1) Extraction of Lithium from Lithium-Brine;
- 2) Processes involved in Recycling of lithium Batteries; **respectively.**

Material Balance: Extraction of Lithium from Lithium-Brine



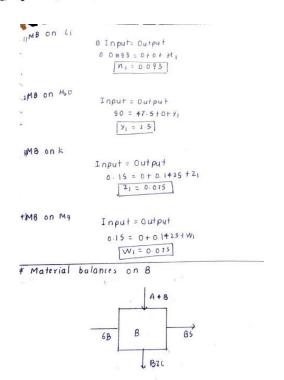
11 - Brine (fresh feed)

- 1) H20 50 kmoles/s
- 21 Li 0.093 Kmoles/s
- 31 K 0.15 Kmoles/s
- 41 Mg 0.15 kmoles/5
- 5) Boron negligible | Unknown values
 50 assume it to be
 inseprable part of brine 5) Boron - negligible

K Assumptions:

- 1) Steady State.
- 2) Reactions only happen in percipitators
- 3) Boron & sulphate composition is considered negligible because of lack of information (consider thus inseprable part of brine)
- 4) Lithium is not Wasted anywhere in the
- 5) Nazcoz completely reacts with Li.

* Table Kmolews	/A	AL	A3	A4B	85	6 B	B74	(8	91	CIOD	Di	6,
Li	0.093	0	0	и,	D	0	H2	0	0	0	0	
H20	50	47.5	0	41	0	0	42	0	0	4	1/5	1
K	0.15	0	0.1425	2,	0.0075		0	0	0	0	0	10
Mg	0.15	0	0 14 29	W,	0.0075	0	0	0	0	0	0	5
Liz(03	0	0	0	D	0	0	0	0	0	V4	Vs	0
No.	of of ateria	unk inde al b add	pend alar itio	enf lenf nces	= + rela	+2 Urion	+ 1 +7	L=9 = 2	(100	2.0	, V4 vesio	, V3
No	of aterio	unk inde al b add	rnow pend alar itio	ins = lenf nces nal	= + rela	+ 2 Urion Do	+ + 15 = F = → A	L=9 = 2 11	(100	% con	, V4 vesio	, V3
No. No. No.	of ateria of	unk inde alb add bala	know pendalar alar itio	vns = enflentes	reland	+ 2 Urion Do	+ + 15 = F = → A	L=9 = 2 11	(100	% con - 2 =	, v4	, V3
No. No No	of aterial of unof ir	unkindegal bala	known pendalar alar itio	vns = lenfines nces nal	TA A A A A A A A A A A A A A A A A A A	+2 Urion DO	+ + + + 	L=9 = 2 112 L	(100	% con	, v4	, V3



No. of unknowns: 2

No. of indpendent: 2

material balances

No. of additional relations: 0

OF: 2-2-0=0

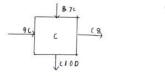
MINB on Li

O+0.093 = G+0+H3

[Nz=0.093]

0+2.5= @# 0+ y3

* Material balances on C



DOF

No. of unknowns=Z
No. of independent MB=1
No. of additional relations=1;
(100% convesion in the read

MB on H_{20} $2.5 + 0 = 0 + y_{+}$ $y_{+} = 2.5$ Reaction

 $2 Li + Na_1 CO_3 \longrightarrow Li_2 CO_3 + 2 N_a$

0.093

med and

Then from above reaction the amount of Na₂(03 need to be add is 0.046

.. Lizto3 =) V4 = 0.046

* Moterial balances on D



00.5

No. of unknowns = 2 No. of independent MB=1

No. of additional relations=1

(Mole tractions are provided)

MB on
$$Li_{2}(03) = 0.046$$

Mole fraction of Li₂CO₃ = 0.98
$$\frac{V_5}{V_5 + Y_5} = 0.98$$

$$V_5 = 0.98V_5 + 0.98V_5$$

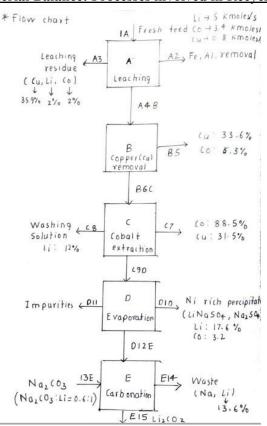
$$0.02V_5 = 0.98Y_5$$

$$V_5 = 49Y_5$$

$$Y_5 = 49Y_5$$

$$Y_5 = 9.38 \times 10^{-5}$$

Material Balance: Processes involved in Recycling of Lithium Batteries



Assumptions:

- 1) Steady state proces:
- 2) The percentange of Li, Co, Cu in all given streams as respect to the moles in the fresh feed.
- 3) The compositions of Li, (o, (u in streams A2, D11 & 13E is zero.

* Table			,		,	,		
Kmoles/s	IA	A 2	A3	AfB	85	BEL	C 7	68
Li	5.0	0	6.1	и,	0	¥2	0	0.6
Co	3.4	0	0.07	У	6.21	y _Z	3	0
Cu	0.8	0	0.29	Zi	0.27	72	0.25	0

Kmoles/s	(90	DIO	DII	DIZE	13E	E14	E15	
Li	Из	6.98	0	И4	0	0.68	и5-	7
Lo	У 3	0.12	٥	4	0	0	0	
Cu	73	0	0	24	٥	0	0	

of Liz (03

Degree of freedom on the whole process

No of unknowns = 14 (M, y_1, Z_1, M_1/y_2, Z_1, M_3, y_3, Z_1, M_4, y_4, Z_4, M_5, Na_1(03))

No of independent = 3+3+3+3+1=13

material balances

only for lithium

No of additional = 1 (Na_1(03 L1 = 0.6:1))

relations

DOF = 14-13-1 = 0

Hence we can solve this system

* Material bolances on A

IA

A3

A1A

A2

Degree of freedom:
No. of unknowns=3 (M1, V1, Z1)
No. of indpendent material balances=3
No. of additional relations=0
DDF=3-3-0=0

IJMB for Li
Input = output
S.0=01+0+X1
M1=4.9

Input = output

3 +0 = 0+007+y,

3 33 = y1

Input = Output

0 8 = 0 + 0 29+ 2,

2, = 0 51

No of unknowns = 3 (H2, Y2, Z2)

No of independent material balances = 3

No of additional relations = 0

DDF = 3-3-0 = 0

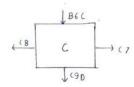
3) MB for (u

Input = output

$$0.51 = 0.21 + 22$$

 $\boxed{2_2 = 0.24}$

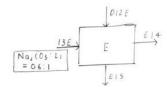
* Material balances on C



DOF No. of unknowns: 3 (n, v, z) No. of indpendent material balances = 3 No. of additional relations = 0 DOF = 3-3-0=0

31 MB for lu

* Material balances on t



No of independent material balances = 1

No of additional relations = 1 (Nazloj Li = 0.6:1) Do F = 2-1-1 = 0

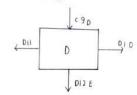
MB for Li

$$3.42 = 0 + 0.68 + H_5$$

$$M_5 = 2.74$$

31 MB for 14

* Material balances on D



DOF No. of unknowns: 3 (n4, y4, z4) No. of independent material balances = 3 No of additional relations = 0 DOF = 3-3-0= 0

Using additional relations

$$\frac{Na_{2}(03)}{3.42} = \frac{0.6}{1}$$

Reaction

But
$$3.42-2\xi_1 = 115$$
 (Remaining lithium)
 $3.42-2\xi_1 = 2.74$
 $\xi_1 = 0.3+$

Hence remaining Naz (03 = 205- &, Na2 605= 1.71

Recovery of
$$Li = \frac{2.74}{5.0} \times 100 = 54.8\%$$
Recovery of $Co = \frac{3}{3.4} \times 100 = 88.5\%$

Strategy/Approach

Throughout the project, we, as a team, changed our strategy several times. In the initial days, we were pretty confused about the project, and therefore, it took a considerable number of attempts to figure out the motive of this course and its project. We decided to choose a scorching topic, i.e., Lithium-ion batteries. At that time, we were not familiar with many factors that could affect our choice of the project. With time, as the course progressed, we started realising the importance of the project. Therefore, we accordingly started to modify our approach. Initially, all of us were working on a single objective at a time.

Nevertheless, very soon, we realised that that kind of an approach would not make enough sense because that would reduce the efficiency and hence, take much more time. So we decided to divide the work amongst ourselves. This time, we randomly allotted one objective to each person. Again, we realised that this was not correct because only one person per objective did not provide any room for discussion. Therefore finally, we decided to divide the work based on the group's needs and everyone's interest in the field. This method finally worked out to be the most effective of all the three.

Moreover, we also increased the frequency of our meetings. This enabled us to update the team at regular intervals. Other than that, we tried to set our virtual deadline before the actual deadline throughout the project. This allowed us to make necessary changes at the last moment.

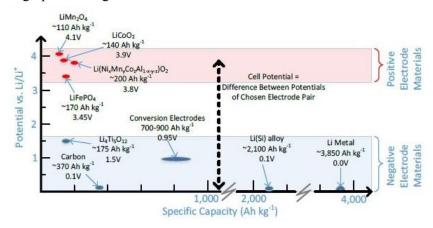
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Sr.No.	Citation	Data used from the research paper
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2.	Lundstrom, M., 2021. Available at: https://doi.org/10.1016/j.jpowsour.2019.227630 [Accessed 21 November 2021].	Values for the material balance of recycling processes.
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4.	Bhave, R., 2021. Available at: https://doi.org/10.1016/j.seppur.2018.05.020 [Accessed 21 November 2021].	Understanding the process of lithium brine extraction.
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7.	Miao, Y., 2021. Energies. Available at:	Understanding the various processes

	http://www.mdpi.com/journal/energies [Accessed 21 November 2021].	involved in recycling LIBs.
8.	Lithium Process Chemistry (2015), Book by Alexandre Chagnes, Jolanta Swiatowska. Available at: <available <a="" at:="" href="http://www.mdpi.com/journal/energies">http://www.mdpi.com/journal/energies [Accessed 21 November 2021].</available>	Various values used in the material balance for lithium extraction from its brine.

Result & Inferences

On doing this project, we found that Lithium-ion batteries usually contain Graphite and Cobalt Oxide as their Anode and Cathode, respectively, because they provide appropriate potential vs specific capacity ratios. Below is the graph showing various Cathode and Anode materials.



However, according to this graph, Li-Metal has the lowest negative potential (0.0V), so apparently, it should have been the preferred material as Anode. Contradictorily, that is not the case. This is because Li-Metal in the free state does not exist because of its highly reactive nature. Next is Li-Si alloy which has V(-ve) = 0.1V. Although Lithium is a very reactive metal, its reactivity drops drastically when forming an alloy with silicon. Therefore, research is being conducted to study the feasibility of using Li-Si alloy as the new Anode.

In the material balance calculations, we observed that almost no lithium gets wasted for the lithium-brine extraction process, and most of the water gets evaporated, thus increasing the concentration of Lithium (98% of the final stream). It is pretty evident that out of roughly 50 kilomoles per second of fresh feed, a very negligible fraction of Lithium is extracted. Also, this process of lithium extraction from lithium-brine takes nearly two years. This is because Lithium is a scarce element, and it is present in a very scattered form. This shows that the current processes of lithium extraction are highly inefficient. Therefore, research on better extraction processes like Direct Lithium Extraction is under observation to resolve this issue.

Whereas, from the material balance calculation of the recycling process, it is evident that the overall recovery percentage of Lithium and cobalt in the recycling process is 54.8% and 88.5%, respectively. This is different from the lithium-brine extraction, where almost 100% of Lithium is recovered. However, these statistics can get quite misguiding. Even though the percentage recovery from the recycling of LIBs for

Lithium is relatively low, the overall absolute mass of Lithium produced in this case is significantly higher.

Future Scope of the Project

More power is required in the world, especially in the form of clean and renewable energy. Our strategy is already being shaped by lithium-ion batteries, which are at the cutting edge of energy storage technology, but what can we expect in the years ahead?

In lithium-ion (Li-ion) batteries, the passage of lithium ions from the positive to the negative electrode via the electrolyte enables energy storage and release. Lithiated metal oxides or phosphates are the most common compounds used as present positive materials. Graphite, graphite/silicon, and lithiated titanium oxides are examples of negative materials.

Key Milestones

Lithium batteries have an energy density higher than any storage technology currently available. The wide range of cell designs and chemistries allows fine-tuning features like quick charging and temperature operating window (-50°C to 125°C). LIBs also have a low self-discharge rate, a long lifespan, and excellent cycling performance, with an average of thousands of charging and discharging cycles.

Major Constraints

Lithium-ion batteries, which contain nickel and cobalt cathodes, are thought to have substantial environmental consequences, including resource depletion, contributing to global warming, ecological damage, and influence on human health. Cobalt is required in large quantities in lithium-ion batteries. LIBs cannot be considered a green alternative to fossil fuels because the extraction of this rare metal is not environmentally or socially responsible.

Area of enhancement and improvement

- A new generation of upgraded Li-ion batteries is deployed before the first generation of solid-state batteries. Solid-state batteries are a technical leap forward. In current Li-ion batteries, ions travel through the liquid electrolyte from one electrode to another (also called ionic conductivity). The electrolyte is replaced with a solid component, ye in all-solid-state batteries, lithium ions can still pass through it. The first key advantage is that solid electrolytes, unlike liquid electrolytes, are non-flammable when heated. Second, it enables the use of cutting-edge, high-voltage, high-capacity materials, resulting in denser, lighter batteries with longer shelf life thanks to lower self-discharge.
- Lithium-sulphur will be the next-generation innovative solution. Sulphur as the positive electrode and metallic lithium as the negative electrode is the active components in a Li-S battery. Because of this, its theoretical energy density is four times that of Li-ion batteries. As a result, it is well-suited to the aerospace and aviation industries.

THE END