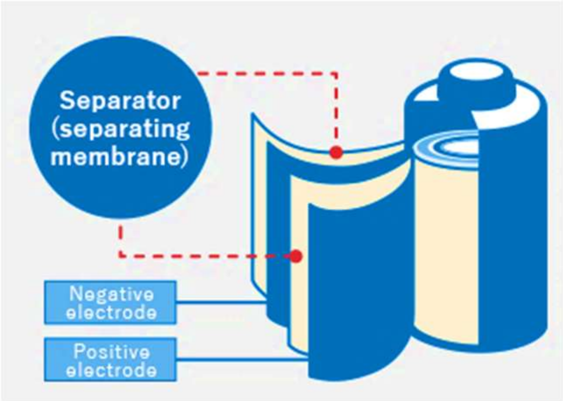
ME 493
BTech Project Presentation

OPTIMIZATION STUDY OF ELECTROSPUN POLYACRYLONITRILE SEPARATOR BY USING CENTRAL COMPOSITE DESIGN



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INTRODUTION

Energy Storage

Renewable energy resources are intermittent in nature. To store the renewable energy, we need energy storage systems.

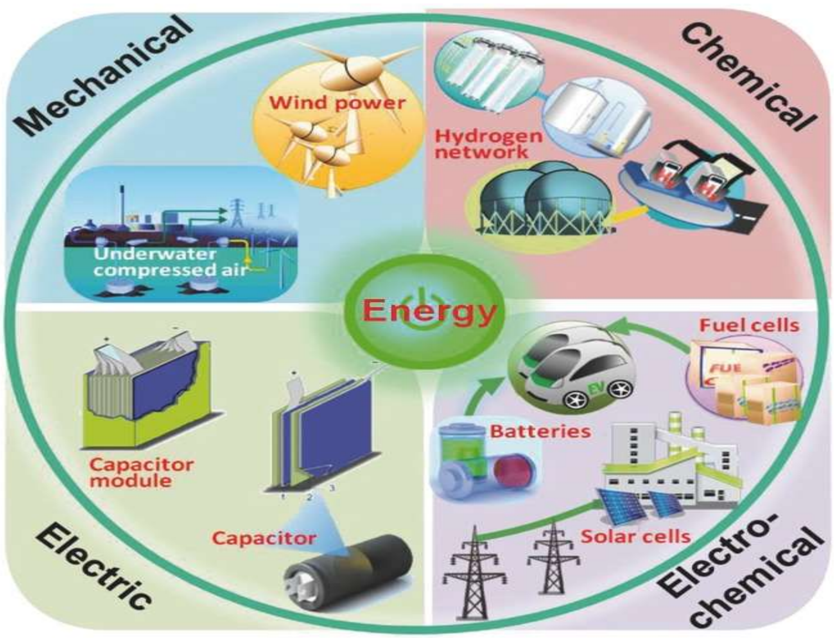


Fig : Various energy storage system [1]

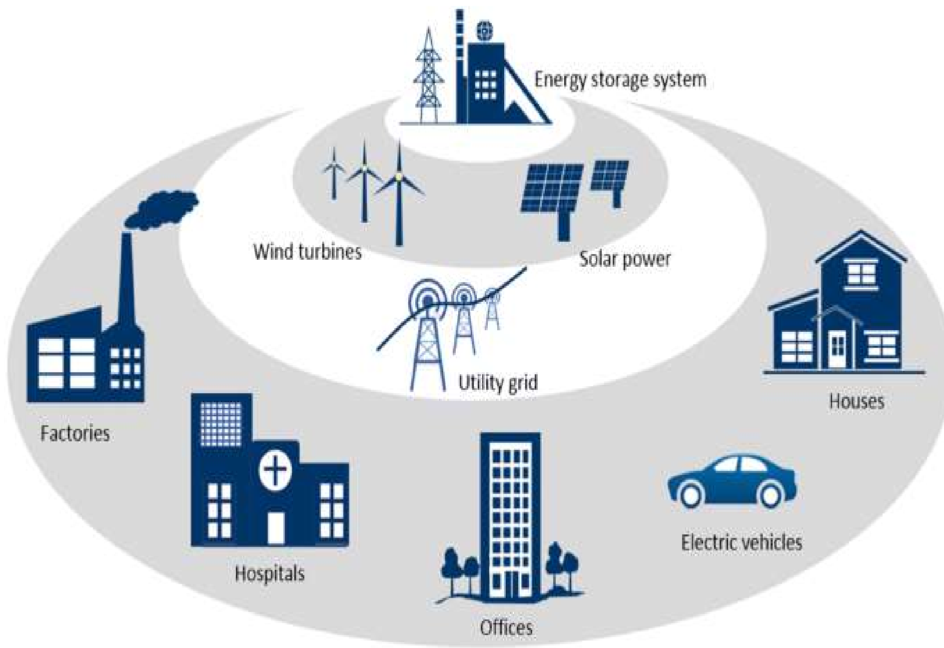


Fig : Applications of batteries [3]

INTRODUCTION

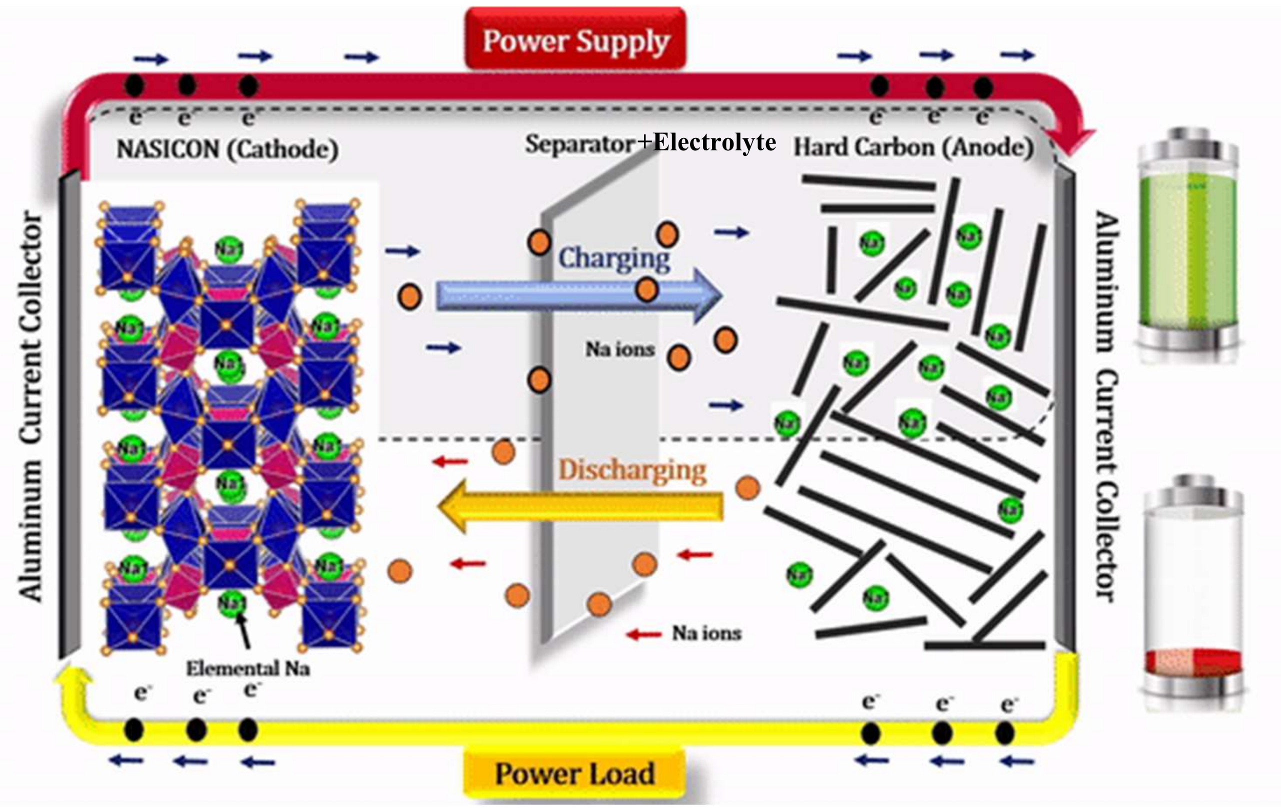
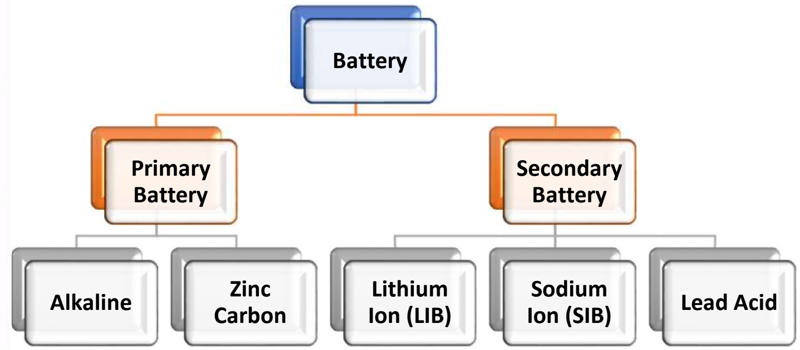


Fig : Charging and discharging mechanism in a SIB cell. [5]



$$NaTMO_2 \underset{Discharge}{\overset{Charge}{\rightleftarrows}} Na_{1-x}TMO_2 + xNa^+ + xe^-$$

$$C + xNa^+ + xe^- \underset{Discharge}{\overset{Charge}{\rightleftarrows}} Na_xC$$

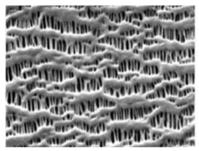
$$NaTMO_2 + C \underset{Discharge}{\overset{Charge}{\rightleftarrows}} Na_xC + Na_{1-x}TMO_2$$

Fig: Equations for working of battery

Separator

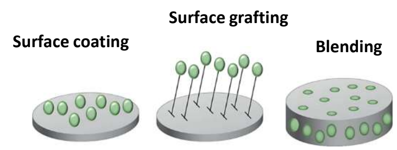
Sr. No.	Parameters	Requirements (LIBs)
1.	Ionic conductivity	10^{-3}-10^{-1} S cm^{-1}
2.	Mechanical strength	>98.06 MPa
3.	Porosity	>40%
4.	Transference number	Near to 1, the better battery performance
5.	Wettability and uptake	Become wet fast and absorb adequate electrolyte
6.	Chemical/electrochemical stability	Long-term stability in batteries
7.	Dimensional stability	Must lay flat with no curl and shrink
8.	Puncture strength	>300/25.4 g μm^{-1}
9.	High temperature melt integrity	>150 ^{o}C
10.	Shutdown effect	Timely and reliable
11.	Wettability	Absorb sufficient liquid electrolyte
12.	Thickness [μm]	<25
13.	Pore size [μm]	<1

Classification of Separator



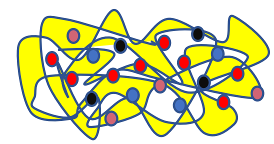
- PP, PE, PP/PE and PP/PE/PP tri layer and
- other polymers

Polyolefin microporous membranes



- Inorganic or organic coating
- Inorganic or organic blending
- Inorganic grafting,

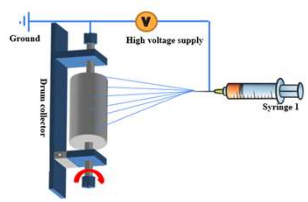
Composite membranes



Electrolyte membranes

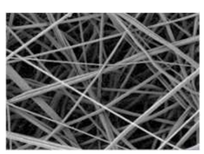
- Solid ceramic
- Solid polymer
- Gel polymer
- Composite

Classification of Separator



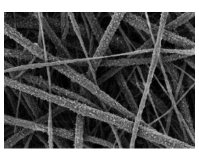
- PVDF, PAN, PVA, PTFE etc.

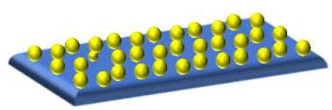
Non-woven separator



FESEM

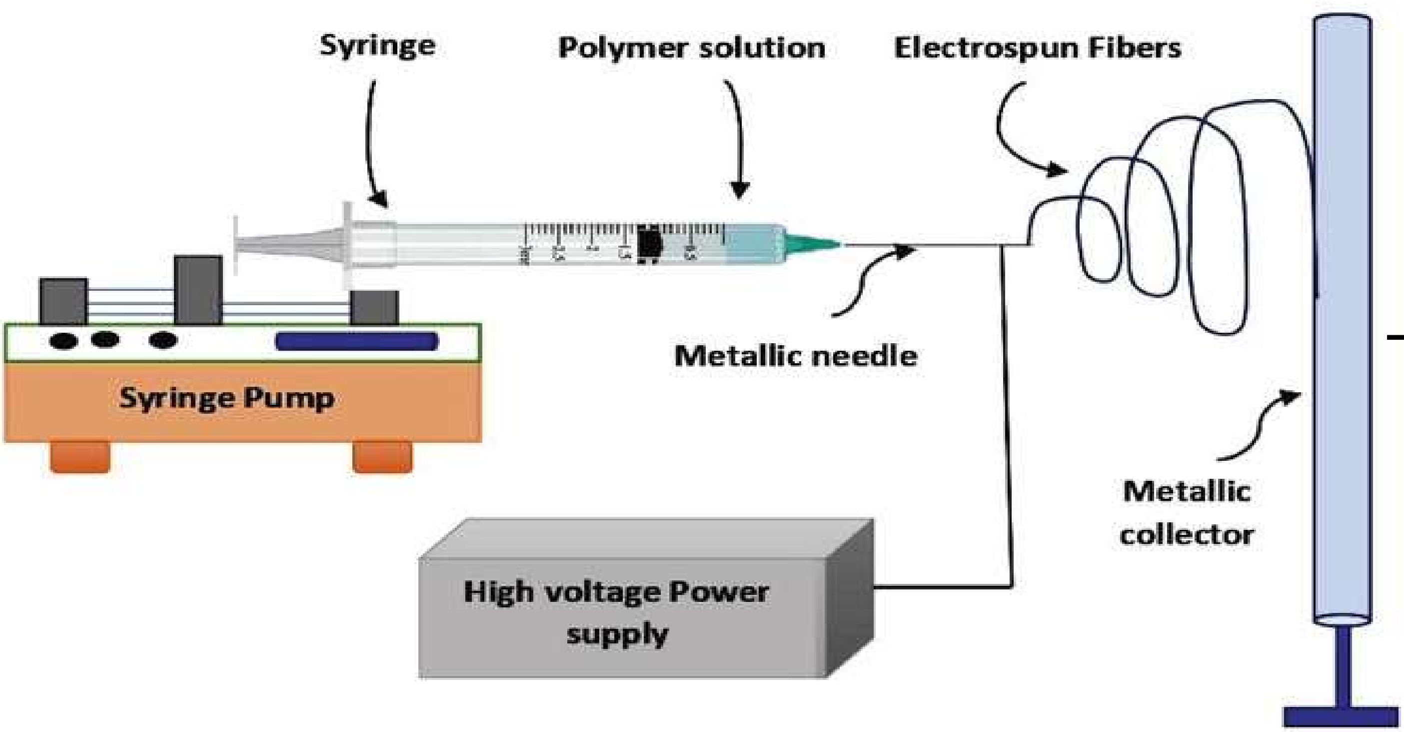
Modified Membranes





- Surface modifications,
- Blending modifications,
- inorganic particle filled modifications

Electrospinning process



Operating Conditions

- ✓ Voltage
- ✓ Solution flow rate
- ✓ Working distance
- ✓ Solution Concentration

Separator

Literature Survey

Property	Polyvinylidene Fluoride (PVDF) [9]	Polypropylene (PP) [8]	Polyacrylonitrile (PAN) [6]	Polyethylene (PE) [7]	Polyether ether ketone (PEEK) [10]
Thermal Stability	High (~160°C – 170°C)	Moderate (up to ~165°C)	High (up to ~300°C)	Moderate (up to ~135°C)	Very high (~340°C)
Mechanical Strength	Moderate	High	High tensile strength	Moderate	Very high
Ionic Conductivity	High (in gel form)	Low	Moderate (can be enhanced with fillers)	Low	Moderate (improved with additives)
Electrolyte Wettability	Excellent wettability	Low	Good wettability with organic and aqueous electrolytes	Low	Moderate
Porosity	High porosity (~70%)	Moderate (~40-60%)	High porosity (~60-80%)	Moderate (~40-60%)	Moderate (~40-50%)
Dimensional Stability	Good	Excellent	Excellent under stress	Good	Excellent
Chemical Compatibility	Stable with organic solvents	Moderate (some limitations with high-voltage electrolytes)	Stable with sodium electrolytes	Moderate (can react with some solvents)	Highly stable with wide range of solvents
Cost	High	Low	Moderate	Low	High
Application as SIB Separator	Frequently used in gel-polymer form	Common, often used in combination with other materials	Suitable, often combined with fillers for enhanced performance	Common, used in multi-layered composite separators	Less common, but very stable and durable

LITERATURE REVIEW

Article	Summary
Zhixin Xue, Dongyang Zhu, Minghui Shan, Hongkang Wang, JiaZhang , GuoshiCui, ZexuHu, Keith, GuiyinXu, MeifangZhu, C. Gordon Functional separator materials of sodium-ion batteries: Grand challenges and industry perspectives. [1]	The operating principle of sodium ion batteries are described. Idealized requirements for sodium ion battery separators are outlined. A summary of existing sodium ion battery separators, including: commercially available separators and their modifications. Industrialization process and future trends of separators are discussed.
Weng, Wei, et al. "Electrospun carbon nanofiber-based composites for lithium-ion batteries: structure optimization towards high performance." [2]	Box-Behnken works well with three-level factors but is less flexible for systems with more complex factor levels. Can require many runs for multiple factors No corner points tested Requires advanced statistical tools and analysis
Hwang, Kyungho, Byeongmin Kwon, and Hongsik Byun. "Preparation of PVdF nanofiber membranes by electrospinning and their use as secondary battery separators." [3]	Developing PVdF nanofiber separators for high-rate discharge batteries via electrospinning. The separators show high electrolyte uptake, mechanical strength, and thermal stability, improving battery performance over commercial options.
WaqasUlArifeen, Minchoel Kim, Jungwook Choi, Kisoo Yoo, Rendi Kurniawan, Tae Jo Ko Optimization of porosity and tensile strength of electrospun polyacrylonitrile nanofibrous membranes. [4]	Taguchi provides limited flexibility in customization of design configurations. Less accuracy for complex interactions. Typically focuses on two-level factors. Less emphasis on interaction effects.

RESEARCH GAP

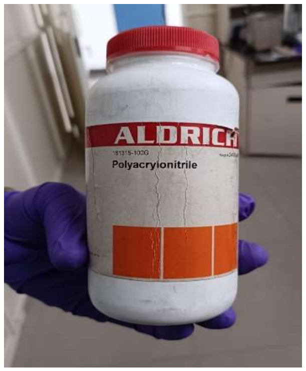
- **Optimizing parameters in electrospinning is technically challenging due to the highly complex and interdependent nature of the process.**
- **Lack of Advanced Predictive Models.**

RESEARCH OBJECTIVE

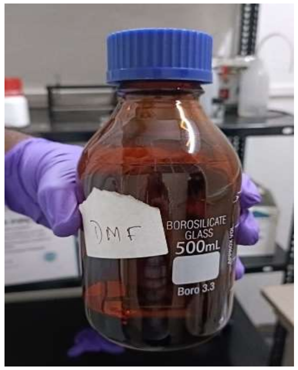
- **Optimizing the electrospinning process using Design Expert software to systematically evaluate and refine key process parameters to achieve desired fiber characteristics by performing minimum number of experiments using central composite design (CCD) on the basis of porosity and fiber diameter.**

MATERIALS

POLYMER Polymer is a long chains or networks of smaller molecules called monomers.



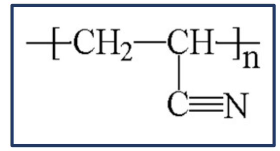
Polyacrylonitrile (PAN)



Dimethylformamide (DMF)

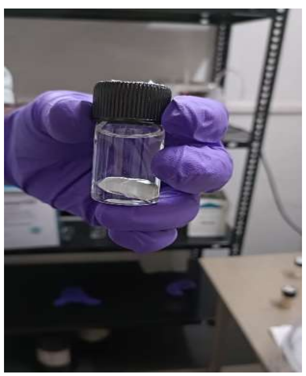
Analytical Balance

POLYACRONITRILE (PAN)



Chemical Formula: $(C_3H_3N)_n$

Melting Point: 300 °C



Polymer Solution

Magnetic Stirrer

Ultrasonic Sonicator

Electrospinning Machine

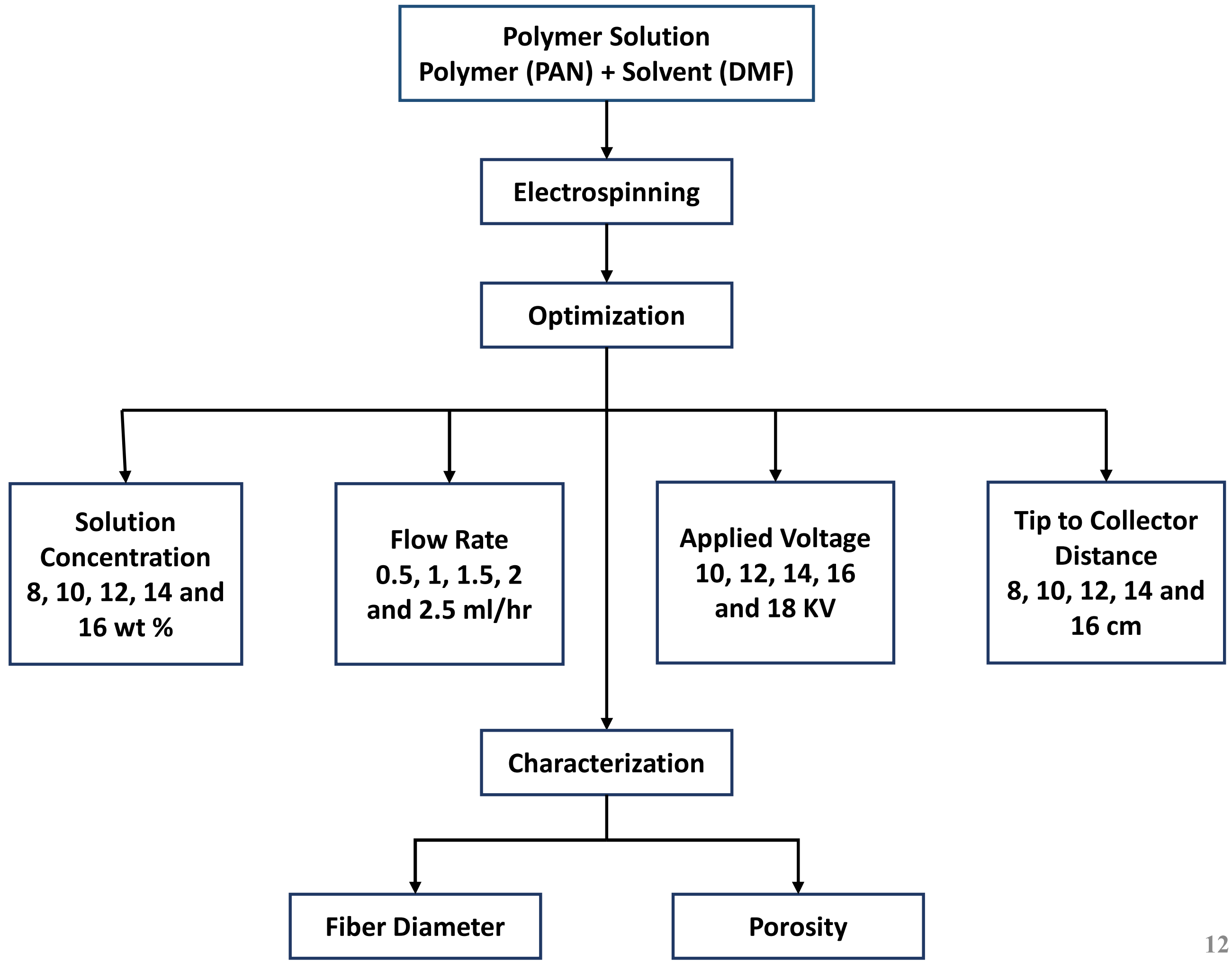
METHODOLOGY

Operating Conditions

- ✓ Applied voltage
- ✓ Solution flow rate
- ✓ Working distance (WD)
- ✓ Collector geometry (e.g., flat or drum)

Ambient Conditions

- ✓ Temperature
- ✓ Relative humidity
- ✓ Atmospheric pressure
- ✓ Air composition (e.g., air, hot air, CO_2, N_2, Ar)

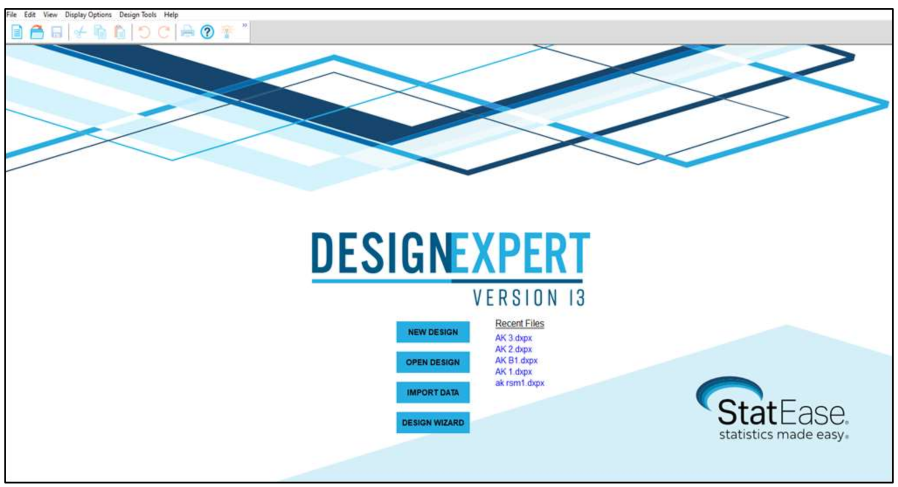


OPTIMIZATION

- Optimization of the electrospinning process requires precise tuning of key parameters to achieve desired fiber diameter, morphology, and material characteristics.
- Some common optimization methods used in electrospinning to improve these parameters.

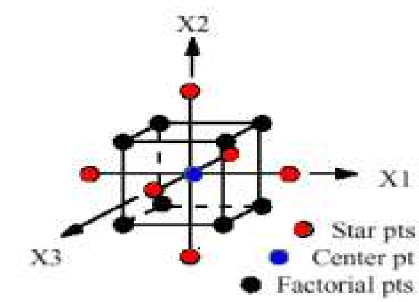
Response Surface Methodology (RSM)

- Response Surface Methodology (RSM) includes various experimental design types, each customized to different optimization needs.
- Design-Expert is an advanced software tool used for designing and analyzing experiments employing a variety of Response Surface Methodology (RSM) techniques.



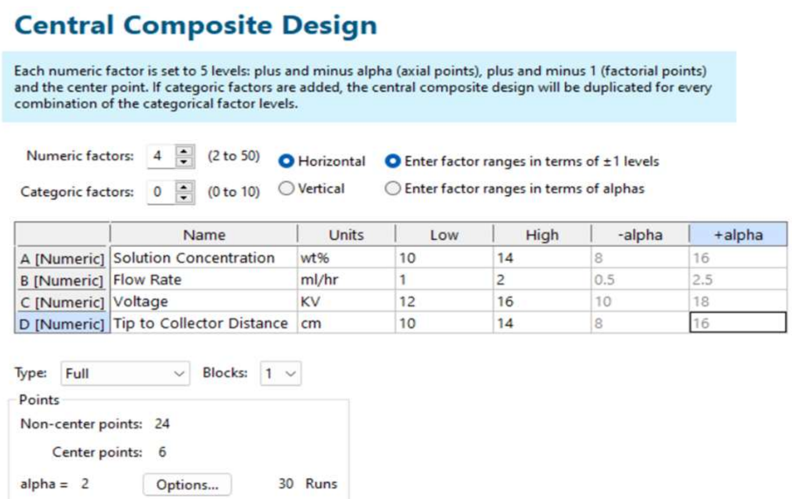
Central composite design

Run #	X1	X2	X3
1	-1	-1	-1
2	-1	-1	1
3	-1	1	-1
4	-1	1	1
5	1	-1	-1
6	1	-1	1
7	1	1	-1
8	1	1	1
9	$-\alpha$	0	0
10	$+\alpha$	0	0
11	0	$-\alpha$	0
12	0	$+\alpha$	0
13	0	0	$-\alpha$
14	0	0	$+\alpha$
15	0	0	0

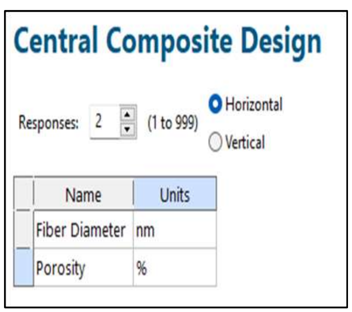


Central Composite Design (CCD)

Central Composite Design (CCD) is a widely used experimental design in Response Surface Methodology (RSM) that helps in optimizing processes with multiple input variables and their interactions.



	Name	Units	Low	High	-alpha	+alpha
A [Numeric]	Solution Concentration	wt%	10	14	8	16
B [Numeric]	Flow Rate	ml/hr	1	2	0.5	2.5
C [Numeric]	Voltage	KV	12	16	10	18
D [Numeric]	Tip to Collector Distance	cm	10	14	8	16

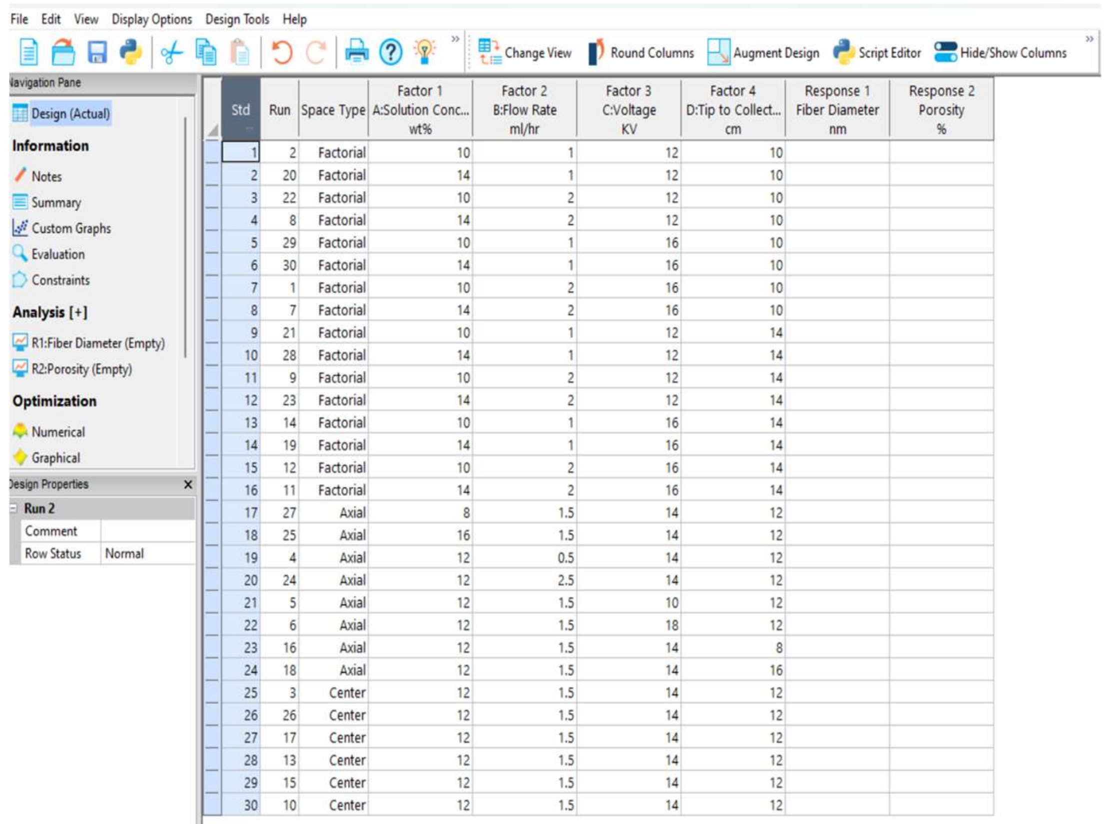


Name	Units
Fiber Diameter	nm
Porosity	%

The porosity was measured by using equation below,

$$Porosity\ (\%) = \frac{M_w - M_d}{\rho_b V_m} \times 100\%$$

where M_d and M_w are the masses of dry and wet membrane after soaking it in n-butanol for 1 h. V_m is the volume of the dry sample, and ρ_b is the density of n-butanol (810 kg/m³).

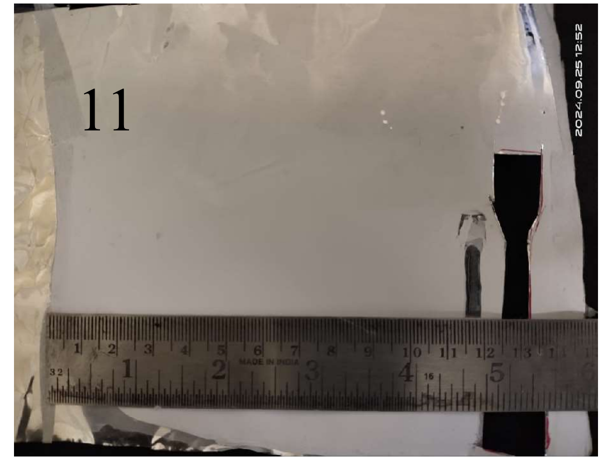


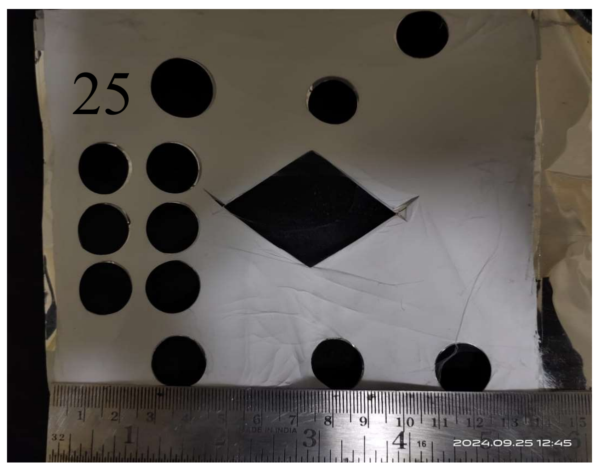
Std	Run	Space Type	Factor 1 A:Solution Conc... wt%	Factor 2 B:Flow Rate ml/hr	Factor 3 C:Voltage KV	Factor 4 D:Tip to Collect... cm	Response 1 Fiber Diameter nm	Response 2 Porosity %
1	2	Factorial	10	1	12	10		
2	20	Factorial	14	1	12	10		
3	22	Factorial	10	2	12	10		
4	8	Factorial	14	2	12	10		
5	29	Factorial	10	1	16	10		
6	30	Factorial	14	1	16	10		
7	1	Factorial	10	2	16	10		
8	7	Factorial	14	2	16	10		
9	21	Factorial	10	1	12	14		
10	28	Factorial	14	1	12	14		
11	9	Factorial	10	2	12	14		
12	23	Factorial	14	2	12	14		
13	14	Factorial	10	1	16	14		
14	19	Factorial	14	1	16	14		
15	12	Factorial	10	2	16	14		
16	11	Factorial	14	2	16	14		
17	27	Axial	8	1.5	14	12		
18	25	Axial	16	1.5	14	12		
19	4	Axial	12	0.5	14	12		
20	24	Axial	12	2.5	14	12		
21	5	Axial	12	1.5	10	12		
22	6	Axial	12	1.5	18	12		
23	16	Axial	12	1.5	14	8		
24	18	Axial	12	1.5	14	16		
25	3	Center	12	1.5	14	12		
26	26	Center	12	1.5	14	12		
27	17	Center	12	1.5	14	12		
28	13	Center	12	1.5	14	12		
29	15	Center	12	1.5	14	12		
30	10	Center	12	1.5	14	12		

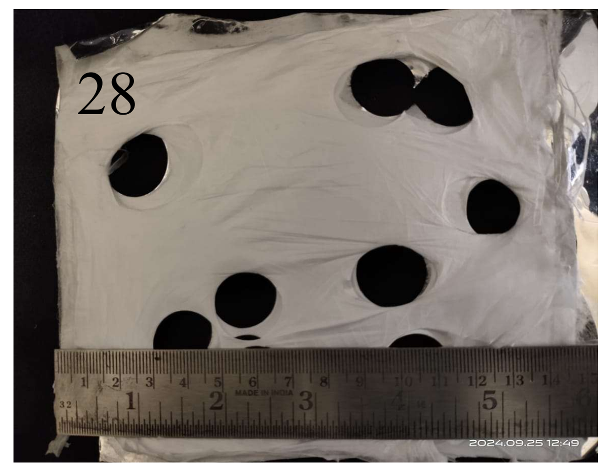
EXPERIMENTAL WORK

Run		Concentration (%)	Flow rate (ml/hr)	Voltage (V)	Diameter (cm)	Results
1	Factorial	10	1	12	10	Good, Continuous & Discontinuous
2	Factorial	14	1	12	10	Not Continuous
3	Factorial	10	2	12	10	Good
4	Factorial	14	2	12	10	Drops are coming
5	Factorial	10	1	16	10	Not Good, Spreading
6	Factorial	14	1	16	10	Multiple Jets, Spreading
7	Factorial	10	2	16	10	Droplet formed
8	Factorial	14	2	16	10	Good
9	Factorial	10	1	12	14	Breaking-Not Continuous
10	Factorial	14	1	12	14	Clogging
11	Factorial	10	2	12	14	Good
12	Factorial	14	2	12	14	Not Continuous
13	Factorial	10	1	16	14	Not Continuous
14	Factorial	14	1	16	14	Multiple jets
15	Factorial	10	2	16	14	Not depositing on rotating drum
16	Factorial	14	2	16	14	Spitting
17	Axial	8	1.5	14	12	Drop of solution falling
18	Axial	16	1.5	14	12	Clogging fast
19	Axial	12	0.5	14	12	Not continuous
20	Axial	12	2.5	14	12	Spitting
21	Axial	12	1.5	10	12	Not continuous
22	Axial	12	1.5	18	12	Multiple jets
23	Axial	12	1.5	14	8	Good
24	Axial	12	1.5	14	16	Breaking
25	Center	12	1.5	14	12	Good
26	Center	12	1.5	14	12	Good
27	Center	12	1.5	14	12	Good
28	Center	12	1.5	14	12	Good
29	Center	12	1.5	14	12	Good
30	Center	12	1.5	14	12	Good

RESULTS AND DISCUSSION







RESULTS AND DISCUSSION

Std	Run	Space Type	Factor 1 A:Solution Concentration w%	Factor 2 B:Flow Rate ml/hr	Factor 3 C:Voltage KV	Factor 4 D:Tip to Collect... cm	Response 1 Porosity %
1	26	Factorial	10	1	12	10	52.5714
2	10	Factorial	14	1	12	10	42.2857
3	3	Factorial	10	2	12	10	47.4286
4	30	Factorial	14	2	12	10	60.8571
5	8	Factorial	10	1	16	10	70.2857
6	28	Factorial	14	1	16	10	75.4286
7	18	Factorial	10	2	16	10	56.2857
8	29	Factorial	14	2	16	10	49.1429
9	19	Factorial	10	1	12	14	57.4286
10	14	Factorial	14	1	12	14	61.7143
11	2	Factorial	10	2	12	14	74
12	22	Factorial	14	2	12	14	47.7143
13	15	Factorial	10	1	16	14	63.4286
14	25	Factorial	14	1	16	14	77.4286
15	7	Factorial	10	2	16	14	71.7143
16	24	Factorial	14	2	16	14	50.8571
17	20	Axial	8	1.5	14	12	56.8571
18	11	Axial	16	1.5	14	12	44.5714
19	1	Axial	12	0.5	14	12	52.5714
20	17	Axial	12	2.5	14	12	70.8571
21	9	Axial	12	1.5	10	12	58.2857
22	13	Axial	12	1.5	18	12	76.8571
23	27	Axial	12	1.5	14	8	40.8571
24	16	Axial	12	1.5	14	16	66.5714
25	6	Center	12	1.5	14	12	42.5714
26	5	Center	12	1.5	14	12	44.2857
27	12	Center	12	1.5	14	12	54.8571
28	23	Center	12	1.5	14	12	39.4286
29	21	Center	12	1.5	14	12	50.2857
30	4	Center	12	1.5	14	12	56.2857

Constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Solution Concentration	is in range	10	14	1	1	3
B:Flow Rate	is in range	1	2	1	1	3
C:Voltage	is in range	12	16	1	1	3
D:Tip to Collector Distance	is in range	10	14	1	1	3
Porosity	is target = 75	39.4286	77.4286	1	1	3

Solutions | Starting Points

Solutions

100 Solutions found

Number	Solution Concentration	Flow Rate	Voltage	Tip to Collector Distance	Porosity	Desirability	
1	**10.000**	**2.000**	**12.000**	**14.000**	**73.905**	**0.969**	**Selected**
2	10.035	2.000	12.000	13.995	73.700	0.963	
3	10.002	2.000	12.000	13.963	73.671	0.963	
4	10.000	1.987	12.000	14.000	73.487	0.957	
5	10.000	2.000	12.058	13.979	73.412	0.955	
6	10.000	2.000	12.000	13.863	73.106	0.947	
7	10.000	1.985	12.000	13.941	73.076	0.946	
8	10.000	1.999	12.144	13.995	72.968	0.943	
9	10.094	2.000	12.000	13.911	72.924	0.942	
10	10.122	1.987	12.000	13.986	72.809	0.938	

Run Order	Actual Value	Predicted Value	Residual	Leverage	Internally Studentized Residuals	Externally Studentized Residuals	Cook's Distance	Influence on Fitted Value DFFITS	Standard Order
1	52.57	63.20	-10.63	0.583	-1.824	-1.997	0.310	-2.363[1]	19
2	74.00	73.90	0.0952	0.583	0.016	0.016	0.000	0.019	11
3	47.43	57.13	-9.70	0.583	-1.665	-1.781	0.259	-2.107	3
4	56.29	47.95	8.33	0.167	1.011	1.012	0.014	0.452	30
5	44.29	47.95	-3.67	0.167	-0.445	-0.433	0.003	-0.193	26
6	42.57	47.95	-5.38	0.167	-0.653	-0.640	0.006	-0.286	25
7	71.71	69.13	2.58	0.583	0.443	0.431	0.018	0.510	15
8	70.29	63.18	7.11	0.583	1.219	1.241	0.139	1.469	5
9	58.29	59.58	-1.30	0.583	-0.223	-0.215	0.005	-0.255	21
10	42.29	47.85	-5.56	0.583	-0.954	-0.951	0.085	-1.125	2
11	44.57	47.35	-2.77	0.583	-0.476	-0.463	0.021	-0.548	18
12	54.86	47.95	6.90	0.167	0.838	0.829	0.009	0.371	27
13	76.86	77.54	-0.6786	0.583	-0.116	-0.113	0.001	-0.133	22
14	61.71	54.33	7.38	0.583	1.266	1.295	0.150	1.532	10
15	63.43	70.81	-7.38	0.583	-1.266	-1.295	0.150	-1.532	13
16	66.57	63.15	3.42	0.583	0.586	0.573	0.032	0.678	24
17	70.86	62.20	8.65	0.583	1.485	1.553	0.206	1.838	20
18	56.29	58.71	-2.43	0.583	-0.417	-0.405	0.016	-0.479	7
19	57.43	56.94	0.4881	0.583	0.084	0.081	0.001	0.096	9
20	56.86	56.06	0.7976	0.583	0.137	0.132	0.002	0.157	17
21	50.29	47.95	2.33	0.167	0.283	0.274	0.001	0.123	29
22	47.71	57.80	-10.08	0.583	-1.730	-1.868	0.279	-2.210[1]	12
23	39.43	47.95	-8.52	0.167	-1.034	-1.037	0.014	-0.464	28
24	50.86	55.52	-4.67	0.583	-0.801	-0.791	0.060	-0.935	16
25	77.43	70.70	6.73	0.583	1.154	1.168	0.124	1.382	14
26	52.57	42.95	9.62	0.583	1.650	1.762	0.254	2.085	1
27	40.86	46.25	-5.39	0.583	-0.925	-0.921	0.080	-1.089	23
28	75.43	70.57	4.86	0.583	0.833	0.824	0.065	0.975	6
29	49.14	52.61	-3.46	0.583	-0.594	-0.581	0.033	-0.688	8
30	60.86	48.52	12.33	0.583	2.116	2.441	0.418	2.888[1]	4

Conclusion

- Literature survey has been completed and material and methods have been finalized.
- Experimental work has been performed with the help of central composite method for optimizing the electrospinning parameters.

Future Work

- To find the average fiber diameter of all the fabricated membranes to know to best morphology by the FESEM and optimize the parameters.
- To optimize the filler percentage to improve the fabricated separator by optimizing electrospinning parameters.

TIMELINE

A

Analysis of topic

Gathered relevant information from various research papers and wrote steps for proceeding

B

Performed the Experiment

Based on CCD Model

C

Result Analysis

through graphs, interaction of various plots

D

Optimization of parameters

A chronological overview of key milestones, steps, and activities that we will complete throughout the duration of the project

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- Department of Mechanical Engineering, IIT INDORE

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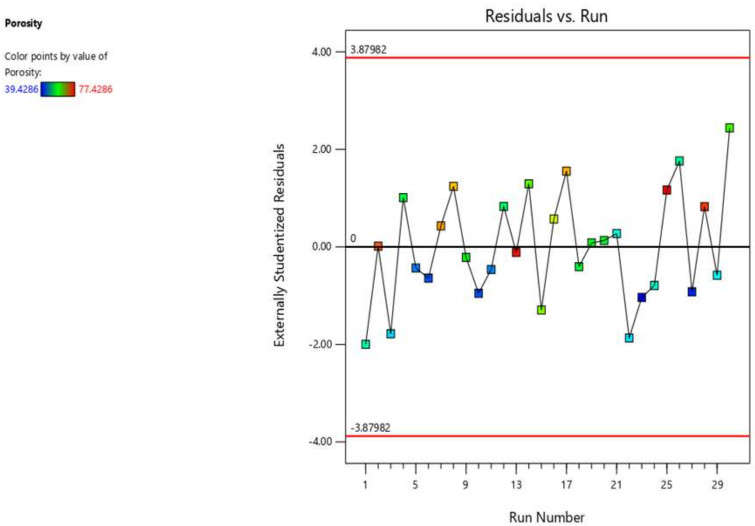
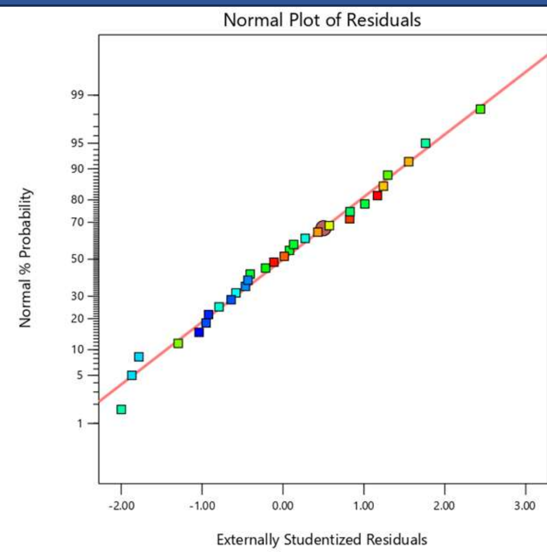
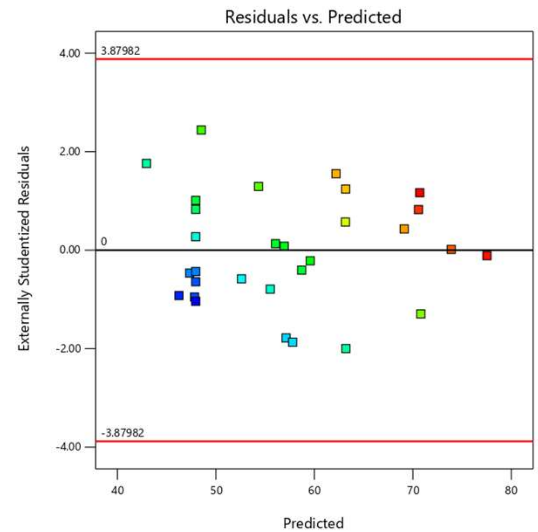
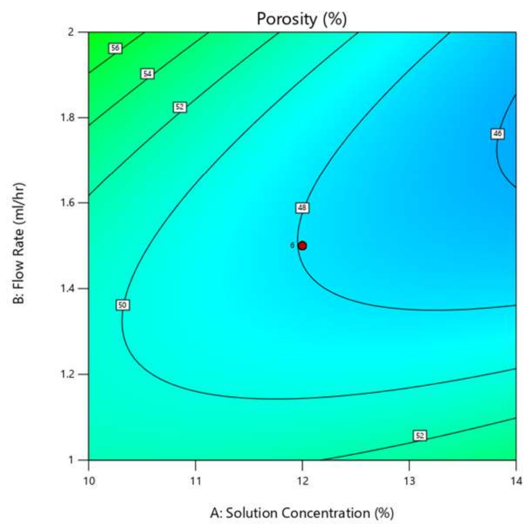
Thank you!!

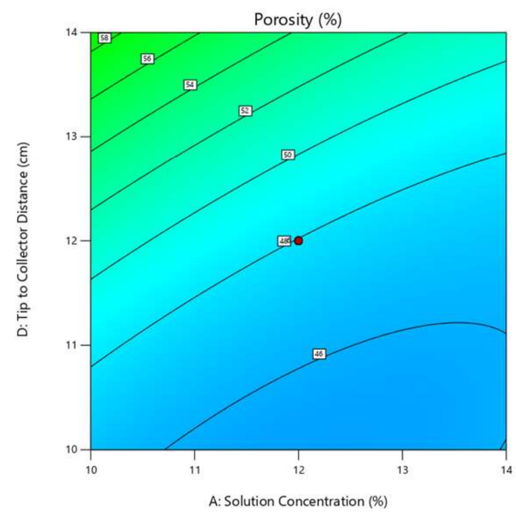
RESULTS AND DISCUSSION











RESULTS AND DISCUSSION