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Tertiary recycling of polyvinylidene fluoride composites by 3D gel printing

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ABSTRACT

In the past, several studies have been reported on the process capability of the material extrusion-based 3D printing process for tertiary recycling of polyvinylidene fluoride composite collected as a waste of dry cell components. But hitherto less has been reported on the process capability of 3D gel printing for tertiary recycling of polyvinylidene fluoride composites. This study highlights the mechanical and dielectric properties of tertiary recycled polyvinylidene fluoride composites processed by 3D gel printing in terms of process capability indices. The process capability indices for mechanical and dielectric properties of tertiary recycled polyvinylidene fluoride composite as dry cell components by gel-printing were observed ≥1, which justifies that the selected process is statistically controlled from a batch production point of view and may be employed as an acceptable process for recycling.

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1. Introduction

Polyvinylidene fluoride (PVDF) is one of the most widely used engineered polymers with tunable piezoelectric properties and can be produced through the polymerization of vinylidene fluoride (Singh et al. 2022). PVDF and its composites have various applications in areas such as biomedical and artificial membranes, filaments for additive manufacturing, batteries, and sensors (Drobny 2008). Few studies have outlined the effect of processing parameters and poling on the electrical and mechanical properties of 3D-printed PVDF sheets using the fused filament fabrication (FFF) process (Porter et al. 2017). Also, studies have been stated on the enhancement of the β phase of PVDF thin films under the effect of stimuli, fabricated using direct ink writing which leads to the improvement of electrical properties of PVDF films in comparison to the solution casting method (Yang et al. 2023). PVDF has been utilized in various energy storage applications due to its high charge storing ability. It is a piezoelectric-based thermoplastic polymer that changes its phase from α to β due to the application of external load (mechanical or electrical) on it. Several studies have been conducted on the improvement of the dielectric and piezoelectric characteristics of poly(vinylidene fluoride-hexafluoropropylene) P(VDF-HFP) and Poly(vinylidene fluoride-tetrafluoroethylene) P(VDF-TrFE) doped with $BaTiO_3$ using screen-printing method (Walkowiak et al., 2007, Carbone et al. 2021). Numerous studies have been stated on the use of PVDF-HFP composite to improve their electrical properties for the application of gate stacks in various electronic instruments (Wu et al., 2006, Xie et al. 2008, Chae et al., 2018). The literature review suggests that different studies have been conducted on the 3D gel printing of PVDF composites for dry cell application. Further, the bibliographic analysis was performed by using data of the past 20 years from the Web of Science database with the selected keywords PVDF, and dry cell. The 92 articles were shortlisted based on relevance criteria from the database. These selected articles were processed with VOS-viewer software (open source) and based on preliminary analysis a total of 3284 terms were noted. Out of these 3284 terms, 121 terms were shortlisted by fixing criteria of minimum number of occurrences as 4. A relevance score was determined for these 121 terms, and the top 60% of terms (73) were considered for analysis (Table 1). Based on Table 1, bibliographic analysis presenting interconnection among various terms is shown in Fig. 1. As observed from Fig. 1, these 73 terms were further subdivided into 4 clusters (cluster 1 contains 22 terms, cluster 2 contains 20 terms, cluster 3 contains 18 terms, and cluster 4 contains 13 terms). Out of these 4 clusters, the term gel electrolyte as a nodal point was selected (Fig. 2) to highlight the research gap.

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Table 1: The relevant terms, occurrences, and their score based on Bibliographic analysis

Id	TermsOccurrencesRelevance score		
1	ANODE	11	0.4611
2	BATTERY	20	0.3326
3	BINDER	10	0.5904
4	CATHODE	16	0.3095
5	COMPONENT	9	1.7952
6	COMPOSITE	10	1.7797
7	CURRENT COLLECTOR	4	1.6923
8	CURRENT DENSITY	10	0.2001
9	DEGREE	7	1.5562
10	DEVELOPMENT	8	0.7432
11	DIFFERENTIAL SCANNING CALORIMETRY	4	0.8308
12	DRY MEMBRANE	5	0.9864
13	DRY STATE	4	3.831
14	EFFECTIVENESS	4	0.8869
15	ELECTROCHEMICAL PERFORMANCE	14	0.4127
16	ELECTRON MICROSCOPY	10	0.8486
17	ELEVATED TEMPERATURE	5	1.4422
18	ETHYLENE OXIDE	4	0.5608
19	FIRST TIME	5	0.7114
20	FOURIER	5	2.2126
21	FTIR	5	2.734
22	FUEL CELL	10	1.9394
23	GEL ELECTROLYTE	6	0.9073
24	GEL POLYMER ELECTROLYTE	7	0.4882
25	HIGH IONIC CONDUCTIVITY	4	0.5007
26	HIGH PERFORMANCE	5	0.3695
27	IMPEDANCE SPECTROSCOPY	7	0.4256
28	INCREASE	9	1.5483
29	IONIC CONDUCTIVITY	12	0.7297
30	LI-ION BATTERY	8	0.6896
31	LIB	6	1.1229
32	LIFEPO4	5	0.4809
33	LIPF6	5	0.3307
34	LIQUID ELECTROLYTE	9	0.8198
35	LITHIUM	5	0.9678
36	LITHIUM-ION BATTERY	11	0.7924
37	LOW COST	4	0.8216
38	MA CM	6	0.4139
39	MA H G	4	1.1187
40	MAGNITUDE	4	1.2885
41	MAH CM	4	1.2838
42	MEASUREMENT	9	0.426
43	MECHANICAL STRENGTH	7	0.5501
44	MIN	4	1.387
45	MORPHOLOGY	10	0.491
46	MPA	5	0.7748
47	MU M	7	0.9032
48	NMP	8	1.3458
49	PAPER	8	0.4225
50	PEO	6	0.4295
51	PORE	4	1.2957
52	POROSITY	10	0.2041
53	PREPARATION	9	0.5501
54	PROTON	5	4.255
55	PROTON CONDUCTIVITY	8	2.9286
56	PVDF BINDER	6	0.7609
57	PVDF HFP	12	0.3086
58	RATE CAPABILITY	7	0.978
59	RESEARCH	7	0.5672
60	ROLE	10	0.6279
61	S CM	7	0.5916
62	SAMPLE	7	1.3685
63	SEM	9	0.7529
64	SEPARATOR	12	0.3556
65	SLURRY	8	1.0431
66	SPECTROSCOPY	14	0.851
67	STEP	7	1.5934
68	SYNTHESIS	4	0.7688
69	THERMAL STABILITY	7	0.9195
70	THICKNESS	8	0.8457
71	VINYLIDENE FLUORIDE CO -HEXAFLUOROPROPYLENE	6	0.6515
72	X-RAY DIFFRACTION	6	1.2118
73	XRD	7	0.8832

Note: In this case study for bibliographic analysis Web of Science database has been explored. However, for future studies, the research gap analysis with other databases may be performed.

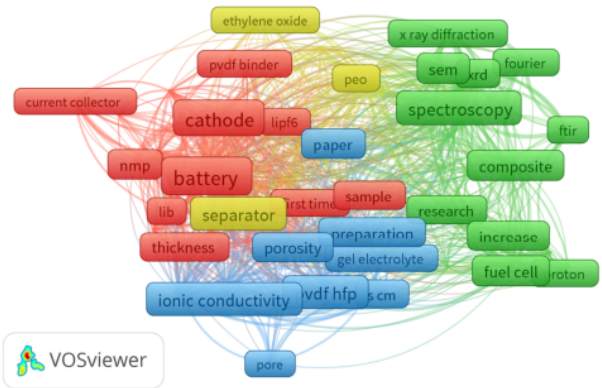


Fig. 1: Bibliographic analysis for the keywords PVDF and dry cell

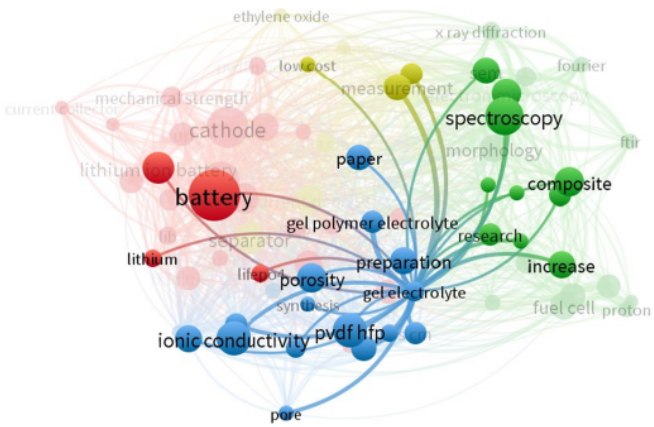


Fig 2: Research gaps (based on Fig 1)

Based on bibliographic analysis it has been ascertained that numerous studies have been conducted on the PVDF-composites for dry cell applications using the material extrusion (MEX) process for tertiary (3°) recycling. But hitherto less has been conveyed on the 3D gel printing of 3° recycled PVDF-composites for the dry cell applications. In this work, the process capability (PC) in terms of process capability indices (C_p and C_{pk}) for modulus of elasticity (E), and dielectric constant (ε_r) of PVDF composites using 3D gel printing has been reported.

2. Material and Methods

Initially, the PVDF granules were mixed with an organic solvent dimethyl sulfoxide (DMSO) in a proportion of 1:6 for the gel preparation. The gel was doped with chemicals/salts (50% graphite, 50% MnO_2, 30% NH_4Cl, and 50% $ZnCl_2$) as the components of the dry cell. The proportions of the chemicals/salts were fixed as per the melt flow index (MFI) value based on the reported literature (Mehta et al. 2023). The entire solution was stirred on a magnetic stirrer at 50°C for 60 min. The PVDF composite gel was further processed on a gel printer (Make-Allevi, USA) having a plastic syringe of 21gauge for the formation of a standard tensile specimen as per ASTM-D638 and a PVDF-composite substrate based on micro-strip patch antenna (MPA). The 3D gel printing of various PVDF composites was performed with a zigzag infill pattern, 4±0.3mm/s printing speed, and 0.170±.003 mm layer height. Further, the 3D gel-printed PVDF composite tensile specimens were tested on a universal tensile machine (UTM) for the evaluation of mechanical properties. The morphological analysis of the cross-section of the tensile-tested PVDF composite samples was performed using scanning electron microscopy (SEM) (Make: Jeol Jsm-6510LV, Japan). Further, the sensing capabilities were explored theoretically using high-frequency simulation software (HFSS) and experimentally using a vector network analyzer (VNA) setup for the 3D gel-printed PVDF composite. After that, the mechanical and dielectric properties of PVDF composites were evaluated for PC using a process capability wizard software. The adopted methodology is shown in Fig 3a. Based on Fig. 3a, the entire process involved in this study is shown in Fig. 3b, which involves sample preparation, 3D gel printing, and testing for mechanical and sensing capabilities. This involves the weighing of PVDF crystals followed by the addition of DMSO solvent, and stirring of the entire solution with a magnetic stirrer for gel formation. The fabricated gel was processed on a gel printer for the fabrication of tensile specimens for mechanical testing and substrates for exploring dielectric properties.

3. Results and discussion

The mechanical properties in terms of peak load (PL), peak strength (PS), and E were ascertained for PVDF composites for the 3D gel-printed standard tensile specimen as per ASTM-D638 and the tensile testing was performed. Further, the morphological analysis of PVDF composites in terms of surface roughness (R_a), amplitude distribution function (ADF) representing the probability of getting the highest peak, peak count (PC), 3D rendered images, waviness, bearing ratio curve (BRC) representing the cumulative probability of getting highest peak, and texture are shown in Fig. 4. As observed from Fig. 4, in all cases (of PVDF and its composites) for ADF normal distribution curve was observed. As regards BRC, PC similar trend was noticed, but for Ra, texture, and waviness profiles the effect of DMSO as solvent is significant.

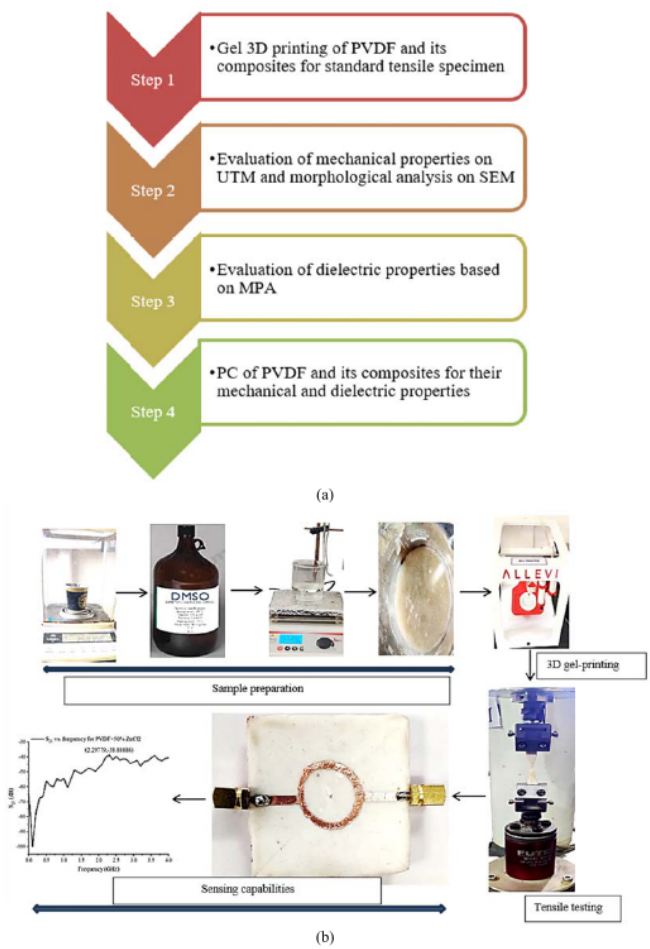
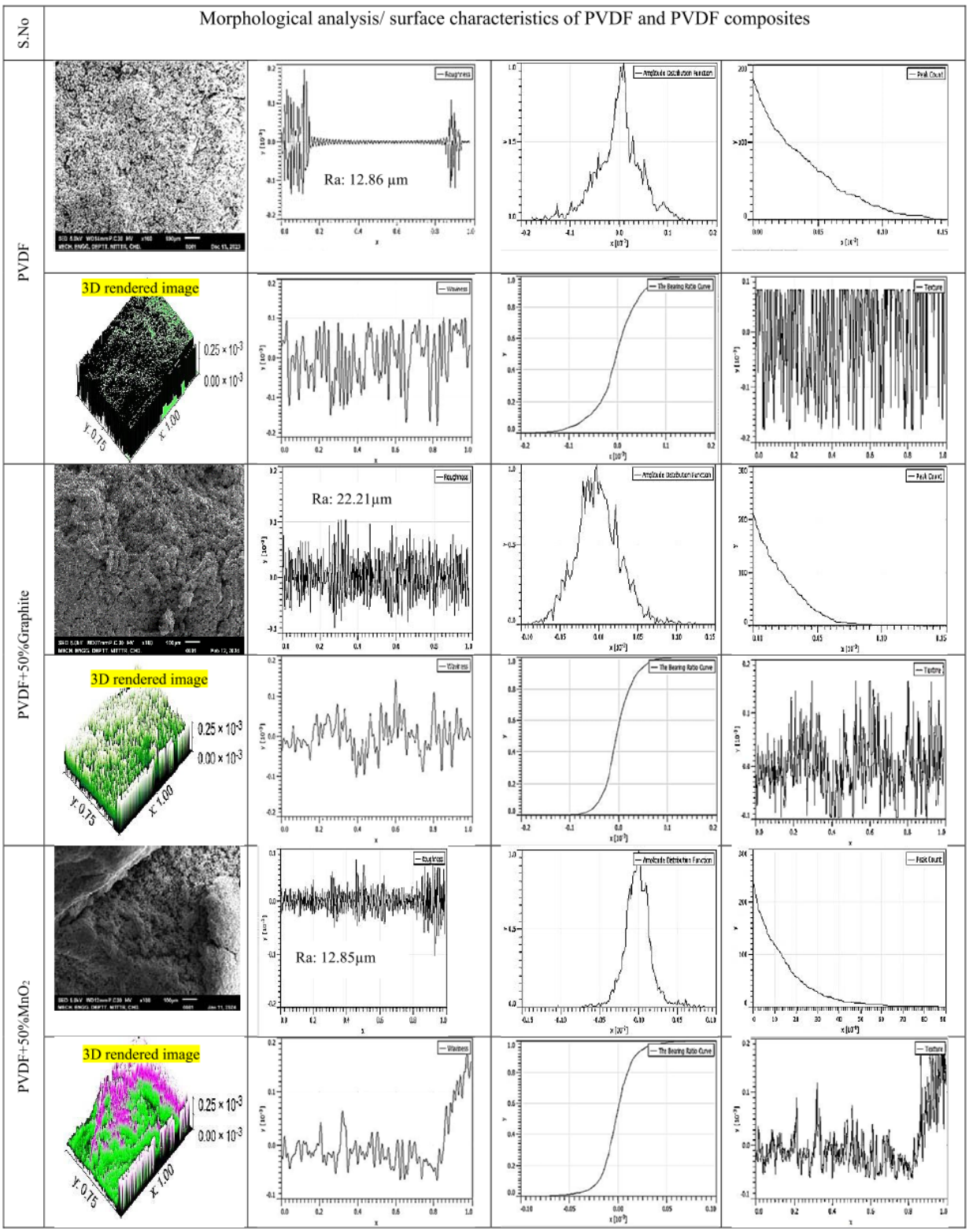


Fig 3: Methodology adopted (a), process involved (b)

Further, the dielectric properties were examined using a ring resonator test as per reported literature (Singh et al. 2022) on the gel 3D printed substrate for different PVDF composites, and its resonance frequency (R_f) characteristics were calculated through a VNA setup, and the results were obtained in the form of insertion loss (S_{21}) (Fig. 5a), and specific absorption rate (SAR) (Fig. 5b). The comparative analysis of mechanical and dielectric properties of PVDF and its composites is shown in Table 3. The PC indices in terms of C_p and C_{pk} for E and ε_r of PVDF and its composites are shown in Fig. 6a, and


S.No
Morphological analysis/ surface characteristics of PVDF and PVDF composites
PVDF
Ra: 12.86 µm
3D rendered image
PVDF+50%Graphite
Ra: 22.21µm
3D rendered image
PVDF+50%MnO2
Ra: 12.85µm
3D rendered image

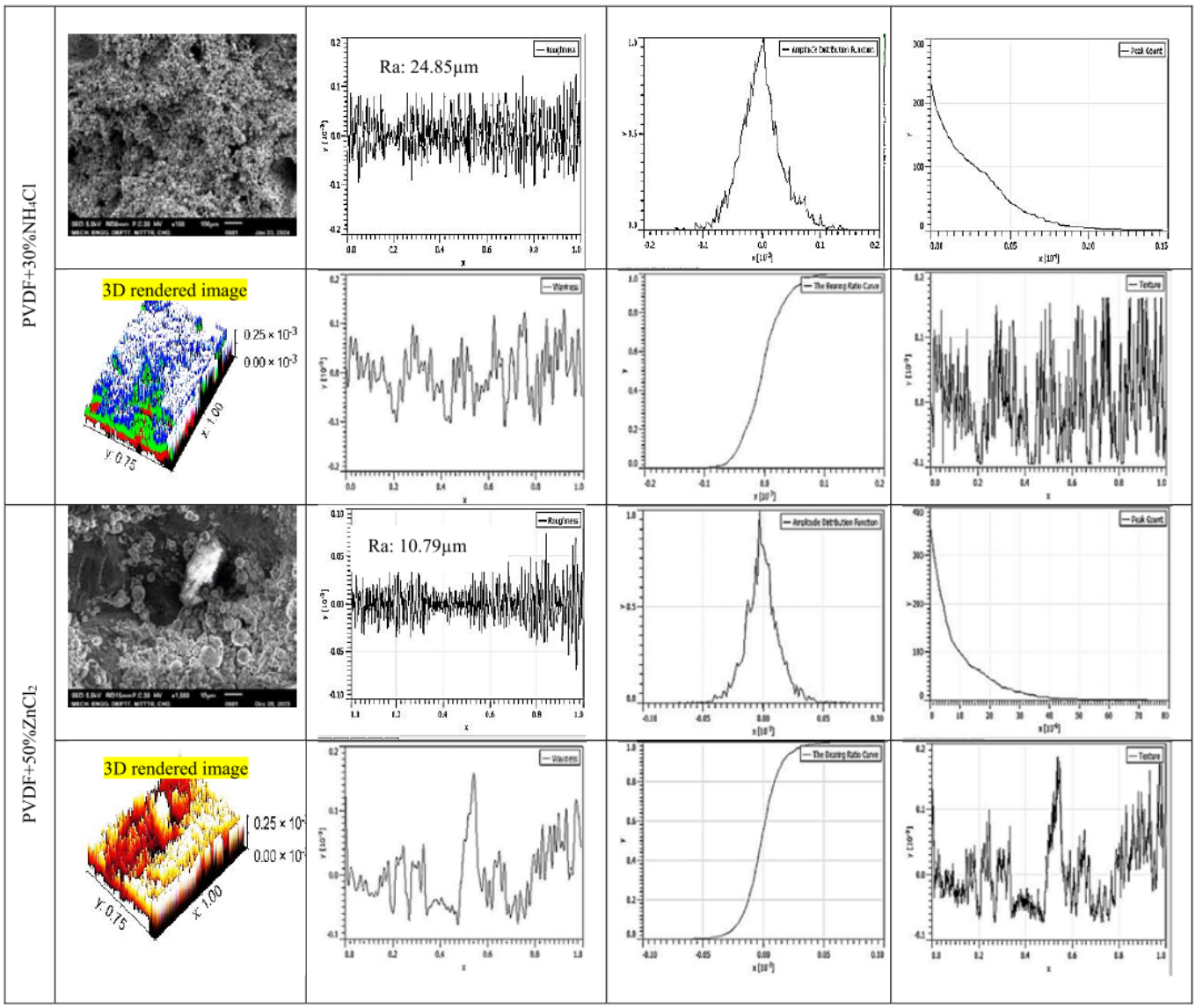


Fig. 4: Morphological analysis/surface characteristics of PVDF and its composites

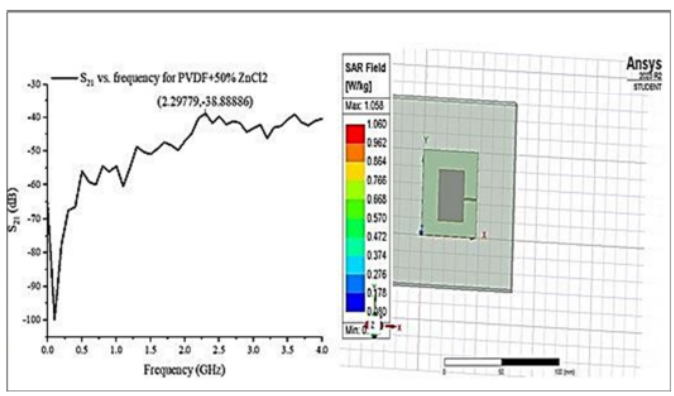
Fig. 6b respectively. Conventionally, C_p and C_{pk} are used with a process that is being control charted. The C_p and C_{pk} indices were calculated based on Eq. 1 and 2.

$$Cp = \frac{(USL-LSL)}{6\sigma} \qquad \text{Eq. (1)}$$

$$Cpk = \min \frac{[USL-\mu)}{3\sigma}, \frac{(\mu-LSL)]}{3\sigma} \qquad \text{Eq. (2)}$$

where USL- Upper Specification Limit, LSL- Lower Specification Limit, and σ=Stanard deviation

The PC results of PVDF and its composites have been expressed in the form of histograms and normal probability curves. The histogram displays the specification limits entered and two bell-shaped curves are theoretical normal distribution curves, having mean = the average of the sample readings. The normal probability plots were prepared for visual checking to ascertain that the data is normally distributed and to find out the nature of the departure from normality. The scattered points shown in the normal probability curve show the variation of data from the true value. As observed from Fig. 6a, and Fig. 6b, for PVDF, there is little deviation of data points from actual data points whereas for PVDF composites the deviation of data



(a) **(b)**

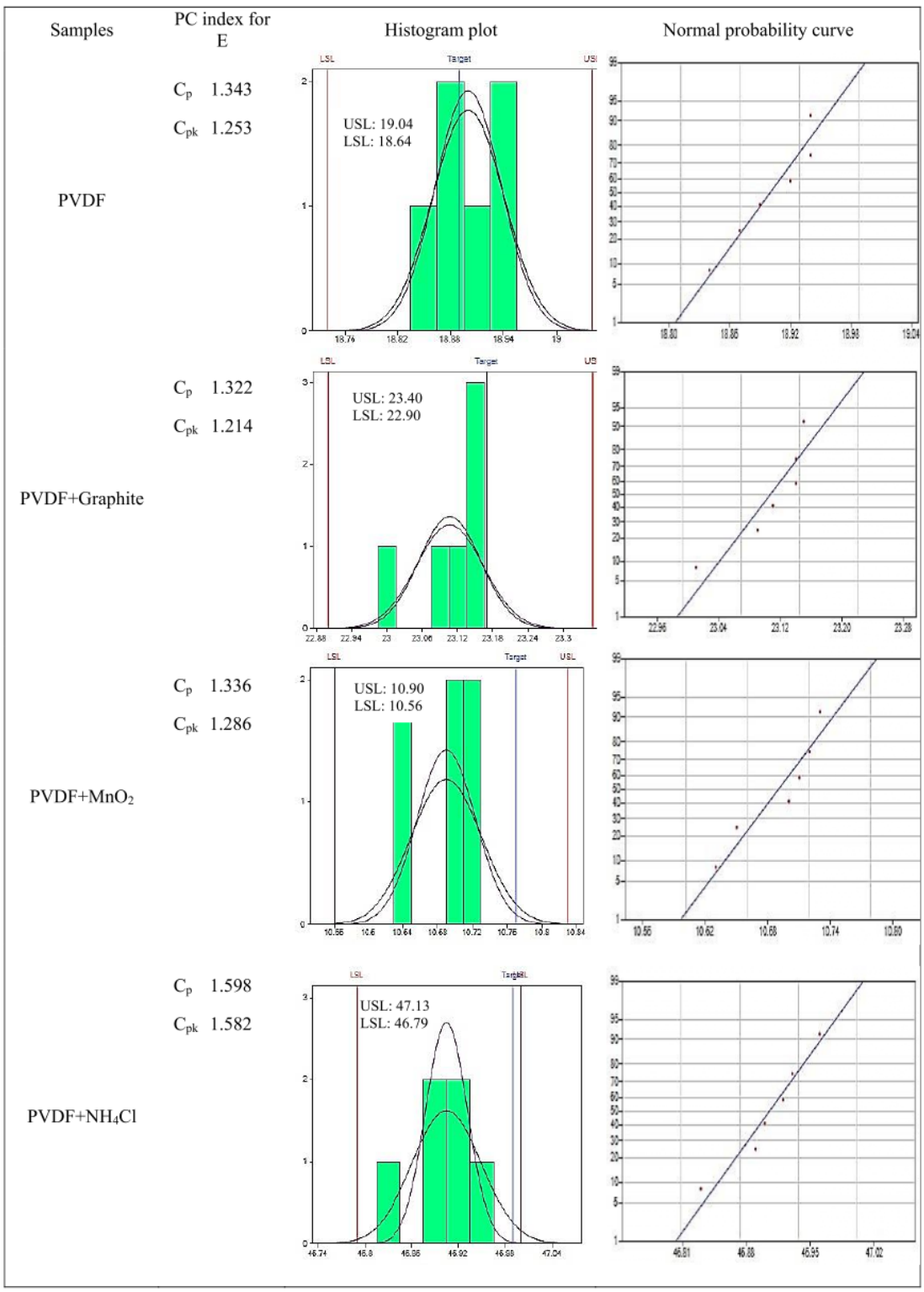
Fig 5: Observed S_{21}parameters (a), SAR value (b)

points is on the slightly higher side. However, for both PVDF and PVDF composites, the process is statistically controlled as $C_p$and $C_{pk}$are ≥1

Table 3: Comparison of mechanical and dielectric properties for PVDF composites

3D gel printing						
Samples	**PL (N)**	**PS (MPa)**	**E (MPa)**	**□r**	**S_{21} parameters (dB)**	**SAR (W/kg)**
PVDF	8.56±0.09	4.82±0.06	18.84±0.20	8.10±0.12	-93.9049	1.175
PVDF+ 50%Graphite	41.43±0.08	20.76±0.05	23.15±0.25	9.10±0.25	-58.3047	1.122
PVDF+50%MnO2	17.81±0.07	8.90±0.04	10.73±0.17	10.97±0.17	-50.7518	1.084
PVDF+30%NH4Cl	9.04±0.06	4.57±0.03	46.96±0.17	9.06±0.22	-38.8888	1.058
PVDF+50%ZnCl2	8.54±0.05	4.27±0.07	21.33±0.05	8.61±0.26	-32.2820	1.459

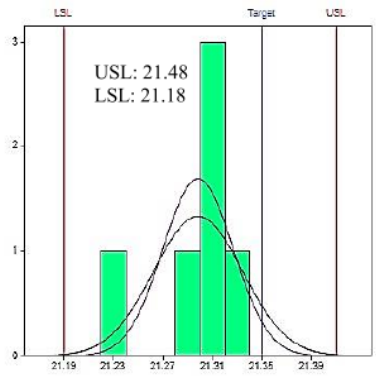
Note: The experiment was repeated 03 times and average values have been quoted.

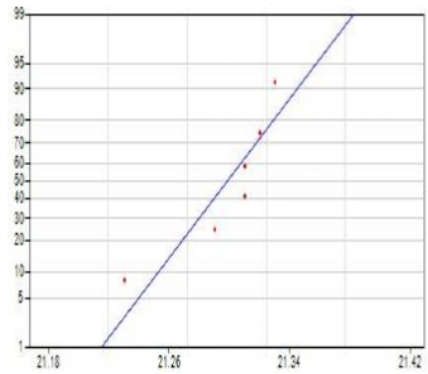


PVDF+$ZnCl_2$

C_p 1.292

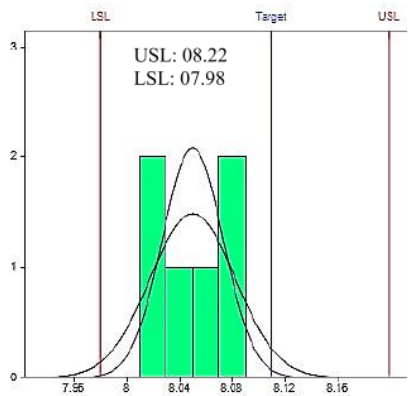
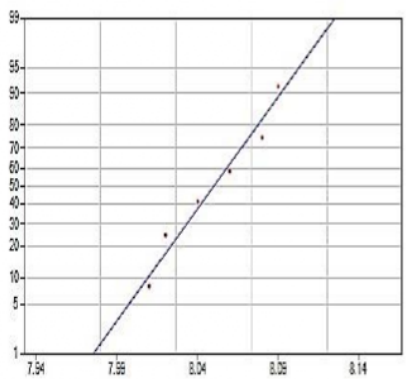
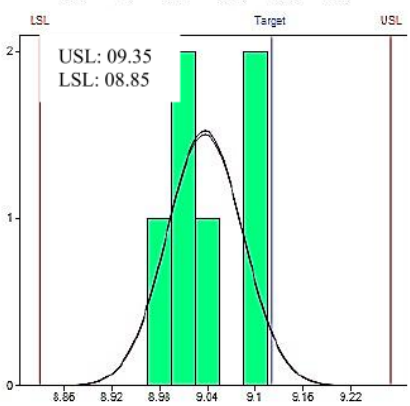
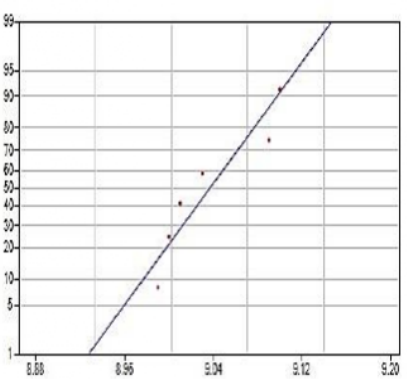
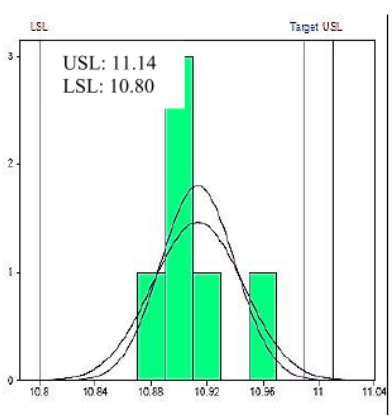
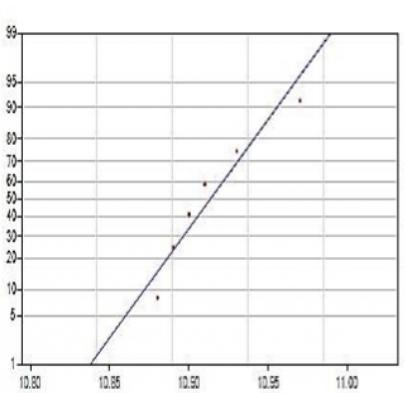
C_{pk} 1.273





(a)

Note: Similar results of PC indices for other mechanical properties (PL, PS) were also noticed.

Samples	PC index for ε_r	Histogram plot	Normal probability curve
PVDF	C_p 1.591 C_{pk} 1.012		
PVDF+Graphite	C_p 1.532 C_{pk} 1.439		
PVDF+MnO_2	C_p 1.316 C_{pk} 1.212		

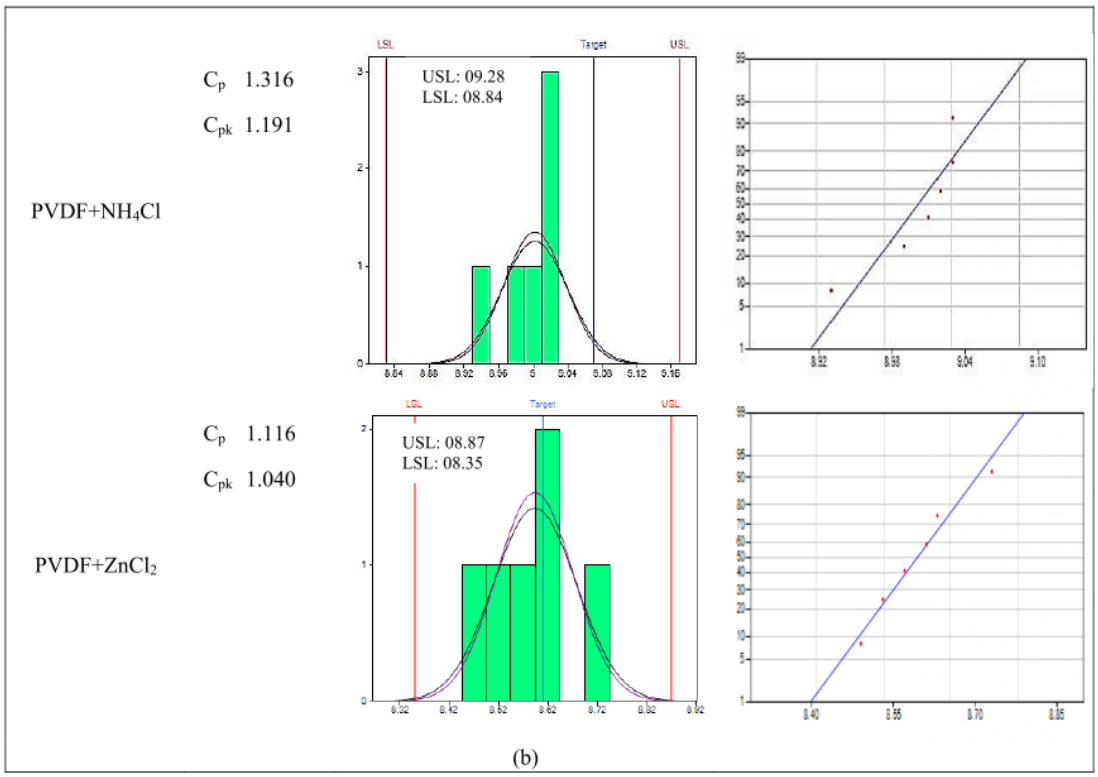


Fig. 6: PC indices for E (a), and □ᵣ (b) of PVDF and its composites

4. Conclusions

The PVDF and its composites have been successfully printed for 3° recycling through gel printing in this study. The mechanical properties (PL, PS, and E) for PVDF and its composites after 3° recycling are in the acceptable range for dry cell application. Based on the C_p and C_{pk} for E, the 3° recycling process is statistically controlled as C_p and C_{pk} are ≥1. The observed value of □ᵣ for PVDF and its composites are in the satisfactory range and based on the values of C_p and C_{pk}≥1 for □ᵣ, the process is said to be statistically controlled. The SAR values of PVDF composites fabricated through gel printing are in the acceptable range (≤1.6) as per the ISM band. Overall, the gel printing of 3° PVDF composites can be explored for commercial manufacturing applications, especially for batch production activities.

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Conflicts of interest: The authors do not have any conflicts of interest/ competing interests.

Consent for publication: All authors have consent for publication of this article.

Author's contribution: Ankush Mehta: Experimentation, data collection, analysis, Rupinder Singh: Analysis, resource generation, B.S.Pabla: Resource generation, validation, B.R.Gurjar: Supervision, validation

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