Multi-Response Optimization for Nano fluids based Mixed insulating fluids using RSM based Desirability Analysis

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Abstract: The present work is aimed to predict the process parameters on a novel insulating fluids using Response surface methodology based desirability function analysis for the sustainability of Transformer oil application. A three factor and three levels Box-Behnken design is used to perform the experiments. Experiments were carried out by adopting a three and three levels Box-Behnken factors design methodology. Analysis of variance was applied to investigate the influence of process parameters and their interactions viz., Weight percentage of ZrO₂ (Wt%), Stirring Temperature (°C) and Weight percentage of CNT (Wt%) on Break Down voltage, Fire Point and Viscosity. With the suitable mathematical models established, a search optimization procedure based on the use of desirability function was used to optimize the performance characteristic. For optimal conditions, a thermal degradation and bonding of cellulose for solid insulation aged in optimized liquid dielectric were characterized through Kraft Paper Surface Texture Analysis.

Keywords: Pongamia Pinnata, ZrO₂, CNT, Breakdown Voltage, Box-Behnken Design, Optimization.

1. Introduction. The demand of electricity in residential, commercial and industrial sector is ultimately increasing especially in fast growing countries. Ultimately the transmission and distribution sectors require power transformers for efficient change of voltage level. For smooth operation of power transformer, it requires efficient transformer oil (Insulating fluid), which decides the efficiency and stability of the transformer. The liquid dielectric (insulating fluid) functions both as electrical insulation and effectual coolant [1]. Since before 1887 petroleum based mineral oil is used as insulating fluids [2].

Notably, the research is focused on enhancing the dielectric strength and cooling of Transformer oil by many researchers. Even though conventional mineral oil has better dielectric strength and good heat conducting properties, alternate liquid dielectric is required owing to high cost, availability and environmental impact. Further many researchers are trying to find sufficient substitution for the insulating fluid of transformer. In general the new insulating fluid should meet the operational requirements [3]. In 1990's usage of vegetable oil is suggested in power capacitors and in transformer as coolant as they are easy and plenty available[4]. In recent year's Silicon oil, Ester oil, Coconut oil and Palm oil were proved to be alternative for Transformer Mineral oil [5,6]. Poor oxidative stability and high viscosity are some disadvantages though vegetable oil has better performance [7]. These vegetable oils offer a wealth of possibilities and play vital role in enhancing the cooling of transformers and increase the breakdown voltage [8]. These oils are biodegradable, have high flash points, high fire points, and lower pour points and have high dielectric breakdown strength. Natural esters insulating fluids like Pongamia Pinnata oils have been focused as alternate for the power transformers [9]. Green oils show better performance provided that proper treatment is done as to meet the standards [7]. Although the natural esters shows good performance, this work is focused on developing mixed green insulating fluids with etherification process. Mixed insulating has better compatibility with the power transformers [5]. Significantly mixed insulating fluids are not mediocre to their pure form and they are environment friendly when compared with mineral oil [10,11]. The mixed insulating fluids which contains Ester aramid and ester cellulose combined dielectrics shows higher break down strength[3]. From the literature the ester based oil and mixed insulating fluids showed better performance. In this paper the work was carried out in two phase. In first phase the preliminary work was done with random proportions to determine the best proportions and in second phase the work was done to determine the

optimum weight percentage of the nano powder to be mixed with the best solutions obtained from first phase results using the response surface methodology approach.

2. Materials and Methods2.1 Materials

In this study, Pongamia Pinnata seeds were obtained from the Shubhasya Biotech, India and crushed to extract oil. Mineral oil was obtained from the transformer oil manufacturers (Manali Lubricants, India) following the standards as a specification. The insulating oils were first pre-processed by filtering the oil through quantitative Whatman filter papers (Pore size: 0.02µm) before blending process. Zirconium Oxide nanopowder (ZrO₂) and Carbon Nano Tubes (CNT) was obtained from Sisco Research Laboratories with purity higher than 96%. The representative Scanning Electron Microscope (SEM) image of ZrO₂ is shown in Fig. 1 (a). ZrO_2 a monoclinic structure, whose dielectric constant is between 23 and 30, with appreciable tensile strength and thermal conductivity. Fig. 1 (b) shows the nano scale microscopic view of CNT. It is highly absorbent, Low thermal coefficient with very high tensile strength.



Fig.1. SEM image of (a) (ZrO₂) and (b) (CNT)

2.2 Pilot Experiments

In this work, the critical properties of novel mixed insulating fluids are explored. These fluids are worthwhile natured and easy obtainable [12,13]. The base Transformer oil and pongamia pinnata oil is taken and moisture content was measured using the Karl fisher iteration method according to ASTM D6304. The magnitude of samples is listed in Table 1, where PO is abbreviated as (Pongamia Pinnata oil) and MO is abbreviated as (Transformer Mineral oil). These samples were stirred well for 5 hrs at 100 °C. Fig.2 shows the samples of mixed insulating fluids which were captured after all stirring process and its shows complete miscibility of blends. Using Magnetic stirrer the heating and stirrer process were carried out. This helps to complete miscibility of mixed fluids and remove if any moisture present.

2.3 Dielectric Test

To determine the electrical seclusion capacity [14] the sample were applied to field of stress using a breakdown test cell in which sphere electrodes with a separation of 2.5mm gap. The test voltage is varied manually to record voltage where the arc is formed between the two electrodes. The sample of 500 ml is poured inside the test cell and three set of measurements were carried out for each samples. The arithmetic average of the three sets of measurement is shown for the Breakdown voltage of liquid dielectric.



Fig. 2. Samples of Mixed Insulating Fluids

2.4 Flash and Fire point Test

Pensky- Martin open cup apparatus was used to find the temperature of prepared samples at which mixture of vapor ignites the flash over and fire on the surface. This determines the safety of the liquid dielectric [15]. Fig. 3 shows the apparatus which is used to determine the flash point and fire point of oil.



Fig.3. Pensky-Martin open cup Apparatus

Category	Sample	Magnitude of oils					
	1	100 % PO					
	2	40% MO + 60 % PO					
1	3	50% MO + 50 % PO					
1	4	80% MO + 20 % PO					
	5	100 % MO					
	6	100 % PO + 0.5 g % Alpha Tocopherol					
	7	100 % MO + 0.1 Wt% of ZrO ₂ (after heating @ 100 °C)					
2	8	40% MO + 60 % PO +0.1 Wt% of ZrO ₂ (after heating @ 100 °C)					
	9	80% MO + 20 % PO + 0.1 Wt% of ZrO ₂ (after heating @ 100 °C)					

Table 1. Proportions of insulating fluids

2.5 Dynamic viscosity Test

Dynamic viscosity is the measure of flow rate of liquid dielectric which indirectly predicts the cooling performance. Redwood viscometer was used to measure the flow rate at 60 °C following the standards [16].

The experiments are carried out following the IEC standards respectively. After accomplishing the experiments, critical parameters like Break Down strength, Viscosity, Flash point, and Fire point were tabulated. Furthermore as an initiative the inter coil insulation Kraft paper's tearing resistance and its stability were analyzed. The degradation point were analyzed using Scanning Electron Microscope (SEM) and techniques [17,18]. IEC 60156 standards was followed for measuring the Breakdown voltage. The Table 2 shows measured breakdown voltage of all samples. Here sample 2 and 8 shows better results, followed by samples 6, 1, 9 & 3. The better results sample has more proportionate Pongamia Pinnata oil in both categories. ASTM D445 standard was followed for the measurement of Viscosity. It is the indirect measure of cooling of Transformer oil as the thermal flow rate is increased due to low viscosity. It's clear here the higher Mineral oil content samples have reduced viscosity. The viscosity is measured at 60°C. The thermal properties are analyzed by its flash point and fire point temperatures. The parameters show sustainability of insulating fluids during high temperature in case of any malfunction. It is clearly observed here that the increased Pongamia Pinnata oil has better flash point and fire point in both categories. In the case of mixed fluids sample 2 and sample 8 has increased flash point and fire point compared to Mineral oils. The overall test results of all samples are tabulated and listed in Table 2.The critical properties as evaluated for all samples infer that the developed mixed fluid has better withstand capability and thermal conductivity.

2.6 Application of Box-Behnken design

In continuation of preliminary experiments the sample 8 was further wide-ranged in addition of the ZrO_2 nano particles with an addition of CNT nano particle. The synthesis was further moved with the runs as listed by design expert software to attain more sustainable and efficient liquid dielectric. Here Response surface methodology based Box- Behnken design was performed

to determine the optimum weight percentage of CNT and ZrO₂ subjected to stirring temperature of solution. A set of 17 experiments according to Box-Behnken with varied weight percentage of CNT and ZrO2 with varied stirring temperature were generated (Table 2) for sample 8(From preliminary experiment). The experiments were conducted randomly to minimize the effects of unexplained variability in the observed responses because of external factors. Response surface methodology (RSM) is a collection of statistical and mathematical technique useful for developing, improving and optimizing process. With the help of RSM the objectives of quality improvement, including reduction of variability and improved performance were accomplished [19-22]. The experiments carried out are shown in block diagram (Fig.4).



Fig.4 Block Diagram for Experiments

Category	Sample	Magnitude of oils	BDV(kV)/ 2.5 mm	Flash Point °C	Fire Point °C	Viscosity at 60 °C/ c-st
	1	100 % PO	52.3	260	290	17.5
	2	40% MO + 60 % PO	55	201	214	13.3
1	3	50% MO + 50 % PO	44.7	185	197	11.8
1	4	80% MO + 20 % PO	37.7	178	195	7.5
	5	100 % MO	30.3	162	178	6.9
	6	100 % PO + 0.5 g % Alpha Tocopherol	53.3	261	290	17.2
	7	100 % MO + 0.1 Wt% of ZrO ₂ (after heating at 100 °C)	31.3	165	180	7.2
2	8	40% MO + 60 % PO +0.1 Wt% of ZrO ₂ (after heating at 100 °C)	56	206	217	14.6
	9	80% MO + 20 % PO + 0.1 Wt% of ZrO ₂ (after heating at 100 °C)	40.7	180	201	8

Table. 2. Test results of Pilot Experiments

In order to improve the electrical stability of the liquid dielectric the process parameters and stirring temperature was optimized using the RSM recently. RSM based Box -Behnken design is a second order technique based on the three level factorial design for three factors and more with the selected points from a system arrangement[23]. The number of run(N) required for this experiment is calculated by N=2k(k-1)+C. Where k is number of factors and C is the enter point. More factors with less run is the main advantage of this design. The three level factors -1(low), 0(Mid Point) & +1(high) should be adjusted to increase the performance. Comparing to other surface design RSM is more efficient, where the efficiency of one experimental design is defined as the number of coefficient in the estimated model divided by the number of experiments. Although, the Box-Behnken design did not combine combinations at lowest or Higher levels of all selected process factors [24].

Design-Expert 8.0 was used to code the variable and to establish the Box-Behnken matrix and to establish the design matrix to analyze the experimental data and to fit the experimental data to a second order design. The coded values of nano particle weight percentage and stirring temperature were determined by the following equation:

$$X_{i} = \frac{x_{i} - x_{0}}{x}, i = 1, 2, 3..; (1)$$

Where X_i the coded value of factor variable, x_i is the actual value of a factor variable, x_0 is the actual value of a factor variable at centre point and Δx is the step change value of a factor variable. The selected three factors are designated as A, B, and C. The response is a function of process a parameters and the relationship is expressed as

$$y = f(A, B, C) + \in ...;(2)$$

The empirical relations were developed to predict all the responses and analysis of variance (ANOVA) was used to confirm the validity of the developed model. Further these functions are incorporated and verified through experiments[25].

The experimental data obtained from this stage were used in multiple regression analysis to develop a quadratic model that relates Breakdown voltage, Fire point and Viscosity. This model is subjected to ANOVA. The optimum weight percentage of nano particle was formulated for achieving increased Breakdown voltage, Fire point and optimized viscosity.

The following quadratic polynomial equation (3) predicts the response with optimized values.

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i=1}^k \sum_{j=i+1}^k \beta_{ii} X_i X_j + \varepsilon; (3)$$

Where Y is the predicted response, β_i are the coefficie nt of the linear terms, β_{ii} are coefficients of quadratic term s, β_{ij} are coefficients of the interaction factors, X_i and X_j in dicated the independent variables and \in is the random nu mber[26]. The values of factors for the development of liq uid dielectric were listed in Table 3 with the levels. The va lues of factors for the development of liquid dielectric wer e listed in Table 3 with the levels.

Table. 3. Parameters and their coded levels

	Ref	Levels			
Factors (unit)	Symbol	-1	0	1	
Wt% of ZrO ₂ (%)	А	0.001	0.0025	0.005	
Stirring Temperature (°C)	В	20	40	60	
Wt% of CNT (%)	С	0.002	0.005	0.008	

		Factor 1	Factor 2	Factor 3	Response 1	Response 2	Response 3
Batch	Run	A:wt% of CNT	B:Stirring Temperature	C:Wt% of ZrO ₂	Break Down voltage	Fire Point	Viscosity
Units		gram	°C	gram	kV	°C	c-st
1	4	0.002	20	0.003	55.2	391.5	15.4
2	6	0.008	20	0.003	11.1	210.6	9
3	7	0.002	60	0.003	18.2	239	9.5
4	11	0.008	60	0.003	33.2	129.1	5.5
5	13	0.002	40	0.001	40.5	476	18.9
6	15	0.008	40	0.001	34.3	406.8	18.1
7	8	0.002	40	0.005	45	334.6	14.6
8	2	0.008	40	0.005	22.9	254.1	10.2
9	16	0.005	20	0.001	36.8	483.4	19.6
10	5	0.005	60	0.001	32.6	154.6	5.6
11	1	0.005	20	0.005	35.7	232.8	12
12	14	0.005	60	0.005	26.1	255.2	10
13	17	0.005	40	0.003	33.4	448.1	19
14	12	0.005	40	0.003	31.2	437	17.4
15	9	0.005	40	0.003	29.8	464.5	21.7
16	10	0.005	40	0.003	32.6	488.3	18.6
17	3	0.005	40	0.003	29.8	461.3	19.7

Table. 4. Experimental observation using Box-Behnken design

3. Result and Discussions

3.1 Statistical analysis and Model Fitting

The statistical analysis was performed using Design Expert Statistical Software package 8.The experimental results in Table 4 were fitted to a full quadratic second order model equation by applying multiple regression analysis for breakdown voltage, fire point and viscosity using the software mentioned above. Based on the proposed second-order polynomial model, the mathematical model is developed from coefficients obtained using the software, which expresses the relationship between Breakdown Voltage, Flash point and Viscosity using the selected parameters (weight percentage of CNT(A), stirring temperature (B) and $ZrO_2(C)$). The final model equations for Breakdown Voltage, Flash point and Viscosity in terms of actual factors (after removing the insignificant terms using backward elimination procedure) as follows:

Break Down voltage =31.549-7.186. A-3.584. B-1.814 . C+14.771. A . B-3.972 . A . C-2.403 . B^2 + 3.880. C^2 (4)

Fire Point = + 448.678 - 55.062.A - 67.55.B - 55.512.C + 87.8.B .C - 66.852.A² - 153.228. B² (5) **Viscosity**=+5.848+2616.082.A+0.9341.B-3962.5.C+75.B.C-326608.A^2-0.0164.B^2 (6)

The significant terms associated with the positive sign coefficient suggested synergist effect and negative sign coefficient indicated an antagonistic effect. Analysis of variance (ANOVA) technique was used to check the adequacy of the developed empirical relationship. In this investigation, the desired level of confidence was considered to be 95% (i.e., $\alpha = 0.05$) and the model adequacies were checked in terms of the values of R², adjusted R², Predicted R², standard deviation, C.V %, Adequate precision and prediction error sum of squares (PRESS).

Table 5 shows that p value for quadratic model is significant which shows that the terms in the model have significant effect on output response. In each Table 5 - 7 indicate that the value of R^2 , R^2 (adj.), R^2 (pred.), called coefficient of determination, is over 80 %. When R^2 approaches unity, the better the response model fits the actual data. From Table 5-7, the model F value indicated that 103.278 for A, 34.750 for B and 18.078 for C imply that the model is significant.

Dieakuowii voltage								
Source	Sum of Squares	df	Mean Square	F- Value	p- value			
Model	1562.06	7	223.151	103.279	< 0.0001			
А	413.211	1	413.211	191.242	< 0.0001			
В	102.784	1	102.784	47.570	< 0.0001			
С	26.347	1	26.347	12.194	0.0068			
AB	872.735	1	872.735	403.919	< 0.0001			
AC	63.127	1	63.127	29.216	0.0004			
B^2	24.392	1	24.392	11.289	0.0084			
C^2	63.578	1	63.578	29.425	0.0004			
Residual	19.446	9	2.160					
Lack of Fit	8.594	5	1.718	0.633	0.689			
Pure Error	10.851	4	2.712					
Cor Total	1581.51	16	R- Squared	0.987				
Adeq Precision	43.551		Adj R- Squared	0.978				
C.V. %	4.558		Pred R- Squared	0.970				

Table. 5. ANOVA table for the Prediction of Breakdown Voltage

There is only a 0.01% chance that a model F value this large could occur due to noise. Lack-of-fit test was also carried out, which measures the failure of a model to represent the data in the experimental domain at points which are not included in the regression. In Table 5 - 7 showed that the lack of fit F value of 0.633 for A, of 4.459 for B and of 1.803 for C imply that the lack of fit is insignificant. There is only a 68.90 % for A, 8.49 % for B and 29.87% for C chance that a lack of fit F value this large could occur due to noise. Adequate precision is used to compare the range of predicted values at the design points to the average prediction error, which is the measure of signal-to-noise ratio. A ratio that is greater than 4 indicates adequate model discrimination. The ratio for the study is 43.55 for A, 18.410 for B and 13.015 for C, which are far greater than 4. Hence, the developed regression model can navigate the response space. The coefficient of variation (CV) indicates the relative dispersion of the experimental points from the predictions of the models. As a general rule, the CV should not be greater than 10% and a high CV indicates that variation in the mean value is high and does not satisfactorily develop an adequate response model. The very low value (4.558 for A, 9.819 for B and 13.440 for v and) of CV clearly representing a very high degree of precision and a good reliability of conducted experiments. The normal probability plot for A, B and C is presented in Fig. 5. The figure revealed that the residuals fall on a straight line implying that the

Source	Sum of Squares	df	Mean Square	F- Value	p- value
Model	239436	6	39906	34.750	< 0.0001
А	24255	1	24255.03	21.121	0.001
В	36504	1	36504.02	31.788	0.0002
С	24653.1	1	24653.1	21.468	0.0009
BC	30835.4	1	30835.36	26.852	0.0004
A^2	18870.3	1	18870.27	16.432	0.0023
B^2	99132.3	1	99132.32	86.326	< 0.0001
Residual	11483.5	10	1148.347		
Lack of Fit	9990.16	6	1665.027	4.459	0.084
Pure Error	1493.31	4	373.328		
Cor Total	250920	16	R-Squared	0.9542	
Adeq Precision	18.41		Adj R- Squared	0.926	
C.V. %	9.819		Pred R- Squared	0.805	

Table 6. ANOVA table for the prediction of Fire Point

errors are distributed normally. Based on statistical analysis, all the results confirmed that the models are statistically significant and there exists a good relationship between the selected factors and response variables.

Table. 7. ANOVA table for the prediction of Viscosity

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Source	Sum of Squares	df	Mean Square	F- Value	p- value
Model	406.322	6	67.720	18.078	< 0.0001
А	30.42	1	30.42	8.121	0.017
В	80.645	1	80.645	21.529	0.0009
С	29.645	1	29.645	7.914	0.018
BC	36	1	36	9.610	0.011
A^2	36.482	1	36.482	9.739	0.010
B^2	183.334	1	183.334	48.943	< 0.0001
Residual	37.458	10	3.745		
Lack of Fit	27.350	6	4.558	1.803	0.295
Pure Error	10.108	4	2.527		
Cor Total	443.78	16	R- Squared	0.915	
Adeq Precision	13.015		Adj R- Squared	0.864	
C.V. %	13.440		Pred R- Squared	0.642	



Fig. 5 Residual plots of all parameters

3.2 Effect of BDV

Fig. 6 depicts the effects of Wt% of CNT and Stirring Temperature on the value of Breakdown voltage under the Wt% of ZrO₂ of 0.0025%. It can be observed that a higher value of BDV is attained in combination of 0.002 Wt % of CNT and stirring temperature of 20 °C. This occurs due to high colloidal stability raised in combination of 0.002 Wt% of CNT and 0.0025Wt% of ZrO₂ presented in mixed insulating fluid [27]. From the same stirring temperature, a Break down voltage tends to decrease from 53.8231kV to 13.4063kV for any value Wt % of CNT.



Fig.6. Effect of Wt% of CNT and Stirring Temperature on Breakdown Voltage

Fig. 7. shows surface plot for Breakdown voltage of mixed insulating fluid in relation with Wt% of ZrO_2 and Wt% of CNT. It can be observed that Breakdown voltage increases gradually with increasing Wt% of ZrO_2 from 0.0010Wt% to 0.005 Wt%. On cantor there is gradual decrease in Breakdown Voltage with

increasing Wt% of CNT from 0.002 Wt% to 0.008 Wt%. Moreover, the charge relaxation time constant of nanotubes in a nanofluid has a major impact on electrodynamics processes occurring in the nanofluid. If the relaxation time constant is long relative to the time scales of streamer growth, the nanotubes will not significantly modify the electrodynamics [28]. However, the nanofluid with concentration of 0.002 Wt% of CNT can be suggested to achieve a voltage of >40kV as the most suitable.



Fig.7. Effect of Wt% of CNT and Stirring Temperature on Breakdown Voltage

Fig. 8 shows the interaction of Wt% of ZrO_2 and Stirring Temperature on Fire Point variation. It is found that the Fire point is decreased with the increased Wt% of ZrO_2 from 0.001Wt% to 0.005 Wt%. On the other hand decrease in Wt% of ZrO_2 with increase in stirring temperature shows gradual increase in Fire point. The high values of both flash and fire points ensure better safety in operations, handling, storage and transportation of vegetable oils and thus better operation safety of transformer using such liquids. The feasible valley region was observed between 20 °C and 40 °C. This is due to the optimized Wt% concentration of nano powders in the liquid dielectrics.



Fig.8. Effect of Wt% of CNT and Stirring Temperature on Fire Point

The viscosity was increased when Wt% of ZrO_2 was decreased from 0.005 to 0.001 Wt% of ZrO_2 but slowly Increases, when the stirring temperature is varied from 60 °C to 40 °C as shown in Fig. 9. However, the viscosity of nanoparticle-oil suspensions increases in accordance with increase in particle concentration in the suspension. Thus particle concentration cannot be increased unlimitedly. It is interesting to note that the rod-shaped particle is favorable to the heat transfer compared with the spherical-shaped particle; however, it has a problem of dispersion instability with the increase in the aspect ratio of particles[29].



Fig. 9. Effect of Wt% of CNT and Stirring Temperature on Viscosity

3.3 Optimization and Model validation using desirability function

From the Fig.6-9 the optimum conditions to achieve high Breakdown voltage with assured higher fire point subjected to proper maintenance of viscosity. This problem is treated as multi-response optimization. The validation of factors is very important to check the response requirement. The individual desirability (d) for each response are obtained by specifying the goals, i.e., minimize, maximize or target the response, and boundaries required for each one. A weight factor, which defines the shape of the desirability function for each response, is then assigned. Weights must be between 0.1 and 10, with larger weights corresponding to more important responses. In this study, a weight factor of 1 was chosen for all the individual desirability values. The best optimized condition of Wt% of CNT is 0.002, Stirring temperature is 20 °C, Wt% of ZrO₂ is 0.0032, Breakdown Voltage is 54.9237kV , Fire Point is 337.27°C and Viscosity is 14.0018 c-st with highest overall desirability value 0.832 is shown in plot (Fig.10).

3.4 Conformation of experiments

In addition of statistical validation, the developed models have also been validated by the confirmation experiments. As from the predicted results the experiments carried out at predicted optimal condition such as Wt% of CNT is 0.002, Stirring temperature is 20 °C, and Wt% of ZrO₂ is 0.0032. All the experiments were replicated four times to obtain average. The experimental Break Down voltage is 52.1kV, Fire point is 380.5 °C, and Viscosity is 13.9 c-st which was very close to the predicted value of Break Down voltage is 55.2kV, Fire point is 391.5 °C, and Viscosity is 15.4 c-st. These results can be considered as good confirmation for the selection of Box-Behnken design to determine the factors for achieving high efficient and stabilized liquid dielectric.

3.5 Kraft Paper Surface Texture Analysis

The SEM focuses high beam energy revealing information about texture, Chemical composition and crystalline structure. It is capable to magnify from 20X to 30000X and spatial resolution from 50 to 100 nm [18,30,31]. The SEM studies were accomplished with JEOL, Japan-JSM 6360. Thermal degradation due to ageing was observed and compared between Typical and Novel oil immersed aged Kraft paper. SEM image of fresh Kraft Paper at 100 micron meter is showed in Fig. 11. It shows cellulose fibers joined mutually and packed in order. As well as, there is no bond breaking between the cellulose fibers. Furthermore cellulose fibers are forming a chain structure[30,32]. Fig. 12 shows an SEM image of ageing Kraft paper of Mineral oil. It was observed cellulose fibers orders are not drastically changed. But some spots appeared as white with slight fiber order displacement and rough surface on samples. This indicates the deterioration of the samples. It is caused by thermal stress. Fig. 13 showed an SEM image of ageing Kraft paper in optimized sample. It makes obvious that when the fibers are tightly packed together, there is no band breaking. When magnification increased, it is clearly indicating that the fibers are packed-in tightly.



Fig.10. Desirability access ramp for numerical optimization in the experiment

Hence, influencing thermal stress on solid insulating material is lower than that of mineral oil immersed Kraft paper[29]. From the SEM analysis, it is found that cellulose insulation degradation is low in the presence of Pongamia Pinnata oil than comparing mineral oil.

The crystallite size of the sample aged with mineral oil immersed cellulosic insulating materials is slightly decreased when compared to new cellulosic insulating materials, since chain scission is very low in crystalline region than that of amorphous, these regions have more free energy when compared to crystal region. Hence, influence of temperature is more in amorphous than that of crystalline region. On the Contrary, the Kraft

paper crystallite size slightly increases whereas pressboard crystallite size is slightly decreased when solid insulating materials are immersed in Pongamia Pinnata oil that of others [11,33]. It infers that degradation rate of solid insulating materials is low when they are immersed in Pongamia Pinnata oil than that of others [32].



Fig.11. SEM image of fresh Kraft paper



Fig. 12. SEM image of ageing Kraft paper of Mineral oil



Fig.13. SEM image of ageing Kraft paper in optimized sample with nano particle trace

4. CONCLUSIONS

In this work, the application of a three factor, three levels Box-Behnken experimental design is helped in reaching a globally optimal solution for enhanced Breakdown voltage, Fire Point and targeted Viscosity level of nano based mixed insulating fluids. The proposed mathematical methodology also provided a critical analysis of the simultaneous interactive effects of independent variables such as Wt% of CNT is 0.002, the Stirring temperature is 20 °C, and Wt% of ZrO₂ is 0.0032.

Then, the study of the effect of nanoparticle on ZrO_2 and CNT was experimentally statistically analyzed. Comprehensive methods and their concentrations for various properties of mixed insulating fluids observed as a function of ZrO_2 and CNT and their concentrations have been reported. The nanoparticle injected into the liquid insulating properties gain the maximum level of improvement.

In particular, the breakdown strength of ZrO_2 and CNT nanoparticle dispersed liquid insulators recorded a maximum improvement. Looking at the dielectric constant and electrical conductivity of the mixed insulating fluid, ZrO_2 and CNT nanoparticle dispersed liquid insulators showed excellent performance.

The properties of mixed insulating fluids have higher flash point and fire point above the rate of enhancement with the increase in the concentration of the volume. Viscosity of the mixed insulating liquid increases because the increase in the concentration of the volume. A detailed study is required to improve viscosity much better in case of mixed insulating fluids.

The Box-Behnken experimental design analysis results showed that the method was indeed robust. Degradation studies of solid insulating materials immersed newly prepared nano based Mixed insulating fluid have been successfully investigated. SEM analysis showed less degradation in solid insulation comparing Mineral oil. All the above studies showed the positive results for using nano Mixed insulating fluids (PO+MO) in the transformer as a liquid dielectric. **References:**

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