International Journal of Mechanical, Industrial and Aerospace Sciences
ISSN: 2517-9950
Vol:7, No:3, 2013

Optimizing Materials Cost and Mechanical Properties of PVC Electrical Cable's Insulation by Using Mixture Experimental Design Approach

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Abstract**—**With the development of the Polyvinyl chloride (PVC) products in many applications, the challenge of investigating the raw material composition and reducing the cost have both become more and more important. Considerable research has been done investigating the effect of additives on the PVC products. Most of the PVC composites research investigates only the effect of single/few factors, at a time. This isolated consideration of the input factors does not take in consideration the interaction effect of the different factors. This paper implements a mixture experimental design approach to find out a cost-effective PVC composition for the production of electrical-insulation cables considering the ASTM Designation (D) 6096. The results analysis showed that a minimum cost can be achieved through using 20% virgin PVC, 18.75% recycled PVC, 43.75% $CaCO_3$ with participle size 10 microns, 14% DOP plasticizer, and 3.5% CPW plasticizer. For maximum UTS the compound should consist of: 17.5% DOP, 62.5% virgin PVC, and 20.0% $CaCO_3$ of particle size 5 microns. Finally, for the highest ductility the compound should be made of 35% virgin PVC, 20% $CaCO_3$ of particle size 5 microns, and 45.0% DOP plasticizer.

Keywords**—**ASTM 6096, mixture experimental-design approach, PVC electrical cable insulation, recycled PVC.

I. INTRODUCTION

WITH the development of the Polyvinyl chloride (PVC) products in many applications, the challenge of investigating the raw material composition and reducing the cost have both become more and more important. PVC is used in many industries including cables, wires, pipes and extruded sheets due to its attractive properties (relatively cheap thermoplastic with a good resistance to acids and alkalis.) Additives including fillers and plasticizers are normally added to PVC compounds to improve its tensile strength, modulus of elasticity, and other properties.

Various components can be added to the PVC. Generally, these additives include antioxidants, stabilizers, plasticizers, antistatic agents, flame retardants, fillers (organic, inorganic, electrical conductive fillers, etc), crystal nucleating agents, etc.

Considerable research has been done investigating the effect of the additives on the PVC; Demir et al [1] studied the effect of adding the ($CaCO_3$ as a filler) on the production of PVC foam, the result shows that the filled samples (containing Ca) had a higher Young's modulus and strength values as compared to unfilled samples. Li [2] investigated the influence of several polymer additives (acrylic K120N, chlorinated polyethylene, PVC-MC100 and polyethylene wax) on thermal decomposition of PVC. The experimental data indicated that polymer additives have important effects on the thermal decomposition, heat release and smoke emission of PVC. Polyethylene wax enhanced the mass loss rate of the carbon backbone of PVC, and shortens its appearance time. This fact can be further confirmed by the behavior and data of heat release and smoke emission in the PVC/polyethylene wax system. Disson and Girois [3] reviewed the use of acrylic process aids in the production of PVC products. Some criteria to optimize the proper selection of the level and nature of the process aids in PVC formulations are given in relation to the final applications, such as films, window profiles, pipes, and so on. Richer [4] reviewed the types, properties, and compounding effect of lubricant on the PVC production and how to choose a lubricant or a combination of lubricants to achieve high quality product, the PVC film was used in the experiments. Van-Den-Oever et al [5] analyzed the effect of a series of colorants for use in polypropylene (PP) and PVC. After processing in PVC at 200°C it has been found that good color intensity is maintained. Also, the best performing natural colorants were found sufficiently heat and light stable for applications where moderate properties concerning heat resistance and light stability are required, such as underground PVC water drainage pipes.

Designed experiments can be used to systematically investigate a process or product variables that influence product response (quality or some performance) and to effectively find the effects of these variables, as well as their levels, that result in optimizing the response. Different approaches fall under the design of experiments (DoE) title

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including factorial designs, Taguchi designs, and mixture designs. In the mixture designs, the variables are proportions of different components of a blend which collectively made up the product under investigation, and the response is function of the proportions of the different ingredients in the mixture. For example, if you want to optimize the tensile strength (response) of stainless steel, the factors of interest might be the proportions of iron, carbon, nickel, and chromium in the alloy. Depending on the mixture experiments type, the response can depend on the relative proportions of the components only, on the relative proportions of the components and the total mixture amount, or on the relative proportions of the components and process variables which are outsiders' factors from the mixture but may affect the blending properties of the mixture.

Most of the PVC composites research investigates only the effect of single/few factors, at a time, on the manufactured PVC product. This isolated consideration of the input factors does not take in consideration the interaction effect of the different factors. In reality, the interaction of the different factors may be more significant than the single factors. This paper implements a mixture-design approach to find out a cost-effective PVC composition for the production of electrical-insulation cables; considering the ASTM Designation (D) 6096 [6].

The next section explains the materials, experimental procedures and testing, and mixture-design approach; utilized in this research. Section three presents mixture-based experiments results and statistical analysis. Finally, concluding remarks are presented in section four.

II. MATERIALS, EXPERIMENTAL PROCEDURES AND TESTING, AND MIXTURE-DESIGNED EXPERIMENTS

A. Materials

Suspension grade of PVC in powder form with a K-value of 70, stearate-acid coated $CaCO_3$ with particle sizes of 5 and 10 microns , recycled PVC from local Jordanian market, di-2-ethylhexyl phthalate (abbreviated as DOP) as a primary plasticizer, Chlorinated Paraffin Wax (CPW) as an optional secondary plasticizer, and a liquid lead heat stabilizer were used in this work. Table I summarizes the materials chosen along with their costs and resources.

TABLE I
USED MATERIALS WITH THEIR COST AND SOURCES

Material	Cost ($/Kg)	Source/comments
PVC Resin	1.4	Georgia Gulf Corporation
PVC Recycled	0.7	Jordanian Local Market
$CaCO_3$	0.11	Jordanian Calcium Carbonate CO.
DOP	3.2	Jordanian Local Market
CPW	1.7	Jordanian Local Market
Stabilizer	3.9	Jordanian Local Market

B. Equipments and Experimental Procedures

The samples were prepared by weighing the amount of each component using a digital balance to sum up to around 2 KGs; then the samples were dry blended using a 6 –Liter high speed mechanical mixer (SHR- 10A model) in order to have a good quality homogeneous Compounds. All samples were mixed till 75˚C and the plasticizer was added to the mix at 60 C to ensure its smooth absorption. The mix is then discharged and cooled to room temperature to avoid material bridging in the extruder feeder which may result in stoppage of materials flow in the extruder barrel.

The dry blended compounds were melt-blended in a counter rotating twin-screw extruder (DSE-20B model) having a 21.7 screw diameter, 850 mm length and 40 L/D ratio. The temperatures settings for the extruder 5 heating zones were 166, 169, 172, 175, 178 ˚C starting from the feeder to the die while the die head temperature was 183˚C. The speed of the extruder was set at 20 rpm. A circular die having a diameter of 3mm was used to produce spaghetti extrudate. The extrudate was then passed through a water bath to cool down, and then cut into pieces for tensile testing. It is worth mentioning that a drawer was used to control extrudate diameter during its water cooling bath.

The Tensile test was carried out using a UTM universal testing machine with a maximum capacity of 5 KN at a cross head speed of 200 mm/min up to fracture, then the autographic record was obtained from which the UTS and the ductility were determined. The tested samples have a circular cross section with a 3 mm average diameter.

C. Design of Experiments by Mixture Design

The mixture-process approach was selected to plan the experiments of this research; where the response is affected by both the relative proportions of the components and the process variables as well.

Four main components (PVC virgin %, PVC recycled %, $CaCO_3$ %, and plasticizer %) and two process variables ($CaCO_3$ particle size, plasticizer type) were selected as summarized in table II which also specifies the lower and upper bounds of each component and process variable levels. These bounds and levels were determined after consulting extrusion experts and based on numerous preliminary experiments. The bounds are expressed in weight percentages of the total compound (excluding the stabilizer percentage). That is, if all the four components are at their upper bound then the virgin PVC weight percentage would equal to 0.3125 (=50/(50+30+55+25).) A fixed percentage of the stabilizer equals to 1.8 % of the total compound mass is added to each mixture as recommended.

The Minitab software was utilized for planning the mixture-design experiments and analyzing the results where the responses/outputs include UTS, ductility, cost and density. It is worth mentioning that 48 runs/experiments have resulted and are shown later in this paper.

TABLE II
BOUNDS OF MIXTURE COMPONENTS

Component	Lower Bound	Upper Bound
A (virgin PVC)	20.0	50.0
B (recycled PVC)	0.0	30.0
C ($CaCO_3$)	20.0	55.0
D (plasticizer)	10.0	25.0
Process Variables	Level 1	Level 2
$CaCO_3$ particle size	5 microns	10 microns
Plasticizer type	DOP only	DOP and CPW*

*In level 2 of the plasticizer type, DOP contributes 80% of the total plasticizer weight and CPW contributes the remaining 20% of the weight.

TABLE III
MIXTURE DESIGN EXPERIMENTAL SETUP AND RESULTED PROPERTIES AND COST

Exp. No.	$CaCO_3$ (percentage, particle size)	Virgin PVC percentage	Recycled PVC percentage	Plasticizer percentage (DOP, CPW)	UTS (MPa)	Ductility (%)	Density (g/cm^3)	Cost (JD/Kg)
1	(20.0, 5)	30.0	25.0	(25.0, 0.0)	7.05	1.80	3.55	0.90
2	(40.0, 5)	30.0	20.0	(10.0, 0.0)	16.23	1.86	2.20	0.80
3	(34.5, 10)	14.5	34.1	(13.44, 3.36)	23.06	1.63	2.86	0.79
4	(34.5, 10)	14.5	34.1	(16.8, 0.0)	27.34	2.73	3.50	0.82
5	(50.0, 5)	20.0	20.0	(8.0, 2.0)	15.70	1.76	2.72	0.83
6	(25.0, 10)	30.0	20.0	(20.0, 0.0)	10.13	1.40	4.11	0.91
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19	(20, 10)	0	55	(20, 5)	3.5	1.63	3.23	0.74
20	(40, 5)	30	20	(8, 2)	14.26	1.85	4.63	0.78
21	(25, 10)	30	20	(20, 5)	9.27	1.85	1.86	0.95
22	(20, 5)	0	55	(20, 5)	3.69	1.71	3.2	0.74
23	(20, 5)	30	40	(8, 2)	13.38	1.82	3.26	0.6
24	(20, 5)	30	25	(20, 5)	9.82	1.25	5.17	0.86
25	(20, 5)	15	55	(8, 2)	12.34	1.93	2.2	0.56
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43	(40, 5)	30	20	(8, 2)	17.36	1.69	3.36	0.8
44	(50, 10)	0	40	(8, 2)	14.57	1.61	3.95	0.77
45	(20, 10)	30	25	(20, 5)	15.8	1.56	3.63	0.88
46	(50, 5)	0	40	(8, 2)	18.23	1.74	3.29	0.75
47	(50, 10)	20	20	(8, 2)	54.11	1.72	0.74	0.85
48	(35, 10)	0	55	(8, 2)	7.95	1.67	2.67	0.62

III. RESULTS AND DISCUSSION

Table III shows a sample of the experiments setup of the mixture design, the properties for the resulted product of each experiment, and the material's cost for producing one kilogram for each composition (expressed in JD where 1JD=$1.4). Note that each of the measured value in this table is an average of three examinations.

Minitab was used to analyze these results. The analysis is conducted in three stages: Analysis of variance (ANOVA), contour optimizer, and response optimizer. The next subsections describe the analysis details.

A. ANOVA

This section introduces the ANOVA for the three studied properties (UTS, ductility, and density) using single replicate (average of three measured values.) Since single replicates are used, the average value for the ANOVA analysis means that there are exactly as many parameters in the ANOVA model as observations. Therefore, in order to test the hypotheses about single and other interactions effects; the four-way and three-way interaction effects between the four components (virgin PVC, recycled PVC, $CaCO_3$, and plasticizer) are neglected and its interaction mean square is used as an error mean square. The F-ratios for all effects (the four main components and their two-way and three-way interactions) are formed by dividing the mean square of the effect of interest by the error mean square. Setting type one error (α) at 0.05, any effect with a P-value less than α indicates significance.

Table IV shows the ANOVA table for the UTS component. Inspecting the P-values for the UTS reveals that the linear component-only model is the only significant model having P-value is less than 0.05. Also, the ANOVA analysis for the UTS shows that $CaCO_3$ particle size and plasticizer type process variables are insignificant.

Residual plots for the ANOVA model were conducted (but are not shown for summarizing purposes). Assessment of these residuals is important to judge the ANOVA model adequacy. The normal probability plot of the residuals and the histogram do not indicate any deviation from normality, thus, normality assumption is appropriate. The plots of the "fitted values" and "observation order" against the "standard residuals" demonstrate the randomness of residuals. Hence, it can be assumed that variability is independent for both "fitted values" and "observation order". Also, from both plots variability can be assumed constant with respect to both variables.

Similar analyses, to the one described for the UTS, were performed for the ductility and density properties. Because of the similarity of their procedure to the previous analysis, the analyses details are not shown here but their results revealed that the ductility can be modeled with linear or quadratic component-only models. Also, the results showed that the plasticizer type is significant in determining the ductility value such that having both the primary (DOP) and secondary (CPW) plasticizers are better for having high ductility values than having primary plasticizer only. Finally, no model showed significance in modeling the density, yet, low $CaCO_3$ particle size and sole primary plasticizer resulted higher density than the other process variables levels.

TABLE IV
ANOVA TABLE FOR UTS

Source	DF	Seq SS	Adj SS	AdjMS	F	P
Regression	26	3570.84	3570.84	137.34	1.62	0.131
Component only						
Linear	3	1717.74	847.63	282.55	3.34	0.039
Quadratic	5	794.29	794.29	158.56	1.88	0.142
Component $CaCo_3$ Size						
Linear	4	27.08	65.87	16.47	0.19	0.939
Quadratic	5	63.71	63.71	12.74	0.15	0.978
Component Plast. Type						
Linear	4	453.34	293.18	73.30	0.87	0.501
Quadratic	5	514.67	514.67	102.93	1.22	0.336
Residual Error	21	1778.39	1778.39	84.69		
Total	47	5349.23				

B. The Contour Optimizer

The contour optimizer accommodates the unique issues surrounding mixture designs. A triangular coordinate system is used which allow to visualize the relationships between the components in a three-component mixture (when the model has more than three components, the components that are not in the plot are held constant. Process variables are also held constant.) In this analysis, six different mixtures of the 4 components were involved, and the sum of each mixture is 100%. The range for the four components are (20-62.5%) for virgin PVC, (0.0-42.5%) for recycled PVC, (20.0-62.5%) for $CaCO_3$, and (10-42%) for the plasticizer. Fig. 1 shows four matrices contour plots for the UTS. Each one of the four matrices has different percentages for the four components. Each matrix has four triangular plots and each one of the four triangles one of the four components is held constant. By analyzing the contour plot it can be seen that matrix 4, has the largest range for the UTS. Note that matrix 4 has the percentage of recycled PVC component held constant, the process variables are 10 microns particle size for $CaCO_3$, and the plasticizer is only DOP. As regarding the mix itself the triangle in matrix 4 with the highest UTS value is the first triangle on the left based on the color index-this occurs where plasticizer value is fixed to 17.5% and the rest will be divided for the virgin PVC and $CaCO_3$. Thus, from the triangle, it can be exemplified that virgin PVC takes 62.5% and $CaCO_3$ takes 20.0% respectively. Therefore, it can be suggested that the mix optimized in the first triangle of matrix 4 provides the maximum UTS.

Similar analysis using the contour optimizer was conducted for the ductility and density properties. The results illustrated that for the highest ductility the compound should be made of 35% virgin PVC, 20% $CaCO_3$ of particle size 5 microns, and 45.0% DOP plasticizer.

C. Determining Optimum (Conditions' Levels) Parameter Settings Using Response Optimizer

The response optimizer in Minitab was used to determine the optimal components percentages and process variables levels that will produce the best output/response values for the UTS, ductility and cost. The response optimizer model was built with the purpose of maximizing UTS and ductility, targeting density, and minimizing cost. Table V shows the model setup. It is worth mentioning that the lower values for the UTS and ductility were selected based on the ASTM Designation (D) 6096 for a product that is used in general purposes (UTS with minimum value of 1500 psi (10.3 MPa).) Also, table V shows the importance of accomplishing each response where minimizing the cost was awarded the highest importance score.

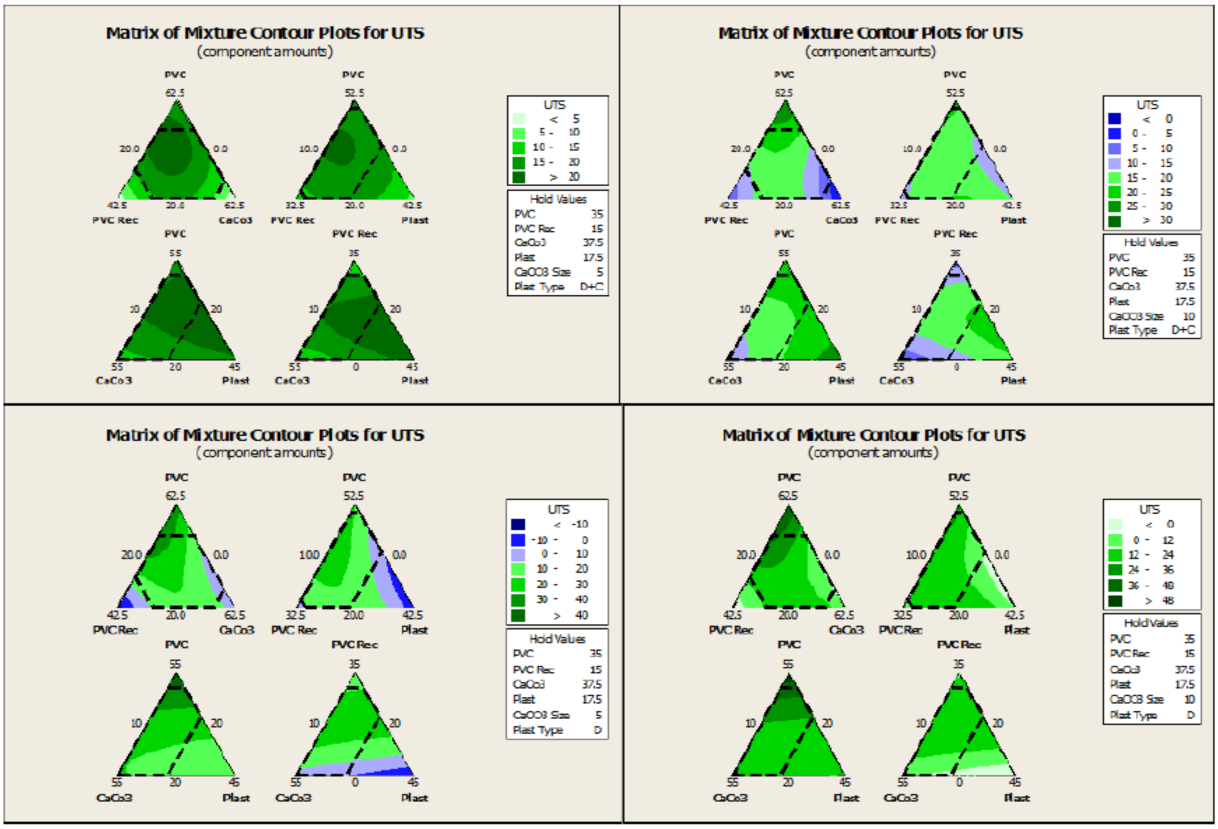

Fig. 1 Matrices of mixture contour plots for the UTS property

TABLE V
THE RESPONSE OPTIMIZER MODEL SETUP

Response	Goal	Lower	Target	Upper	weight	Importance
UTS	Max.	10.3	50.0	-	-	5
Ductility	Max.	1.5	5.0	-	-	5
Density	Target	1.0	1.5	2	2	1
Cost	Min.	-	0.5	1	1	10

The solution of the solver optimizer revealed that local optimum values of UTS, ductility, and cost are 17.32 MPa, 322%, and 0.97 $/kg, respectively. These responses values can be obtained at the component percentages and process variables given in table VI.

TABLE VI
LOCAL OPTIMAL PERCENTAGES/SELECTIONS FOR INPUT COMPONENTS AND PROCESS VARIABLES

Component/process variable	Optimum percentage/selection
Virgin PVC	20
Recycled PVC	18.75
$CaCO_3$	43.75
Plasticizer	17.5
$CaCO_3$ particle size (microns)	10
Plasticizer type	DOP & CPW (80/20 ratio of the plasticizer total 17.5%)

IV. CONCLUSIONS

The effects of virgin PVC percentage, recycled PVC percentage, CaCO3 percentage and particle size, and plasticizer percentage and type; on the mechanical properties of PVC electrical insulation and cost were examined. The DoE mixture approach was adopted in designing the experiments and analyzing the results. The analysis showed that for maximum UTS the compound should consist of: 17.5% DOP, 62.5% virgin PVC, and 20.0% $CaCO_3$ of particle size 5 microns. For the highest ductility the compound should be made of 35% virgin PVC, 20% $CaCO_3$ of particle size 5 microns, and 45.0% DOP plasticizer. Finally, a minimum materials cost for a PVC insulation of an electrical cable can be achieved through using 20% virgin PVC, 18.75% recycled PVC, 43.75% $CaCO_3$ with participle size 10 microns, 14% DOP plasticizer, and 3.5% CPW plasticizer. This minimum-cost composition will result in ASTM acceptable values of 17.32 MPa for the UTS, 322% ductility, and it will cost 0.97 $/Kg under the Jordanian market conditions.

ACKNOWLEDGMENT

The authors would like to acknowledge the Jordanian Scientific Research Support Fund (SRF) for supporting funds required for this study under the fund number H/2/05/2009.

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