

Optimization of Tensile Strength of Hollow Polyvinyl Chloride Wire Insulation by Factorial Experimental Design

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Abstract—With the development of Polyvinyl Chloride (PVC) the need of proper governance of additives and filler materials have become very important. The effect of additives, mainly plasticizer and filler on the ultimate tensile strength of Polyvinyl chloride (PVC) compounding is examined. The inner diameter and outer diameter for each of the sample were taken as 1.5 mm and 2.7 mm respectively. The mixtures were prepared by varying the composition of the constituents. The ingredients used were PVC Resin (40%-50.00%), Calcium Carbonate (19.34%-29.82%), Chlorinated Paraffin Wax (8.70%-12.04%) and Di-octyl Phthalate (9.65%-19.44%). This paper implements an experimental design approach to find out PVC composition for the production of electrical-insulation cables considering IS: 5831 – 1984 standards. Experimental data analysis was done by ANOVA (Analysis of Variance), Surface Plots, Scatterplot Graphs and Response Optimizer.

Keywords: PVC compounding, ultimate tensile strength, hollow wire insulation, experimental data analysis

1. INTRODUCTION

With the development in Polyvinyl Chloride (PVC) it has become very important to ascertain the practical effect of composition on its mechanical behavior. Presently there are 50 different basic types of plastics, included in 60,000 different plastics formulations. Those based on polyolefins and polyvinyl chloride, have highest utilization worldwide. Mechanical properties are considerably affected by the type and amount of plasticizer. To a lesser extent fillers will affect the physical properties. Un-plasticized PVC is a rigid material while the plasticized material is flexible and even rubbery at high plasticizer loadings [1]. Depending on how PVC is compounded it may have rubber-like, flexible properties or have high rigidity. The formulation is determined by the future application of the fabricated PVC product, which can be electrical insulation, medical tubing, food wrap, garden hose, flooring and clothing. The formulation is also determined by the processing technique which is employed [2]. Fillers are used for a wide variety of reasons. They can extend resin, increase stiffness and strength, improve impact performance,

and shorten cycle times. They prevent hang-up in dies and neutralize the products of degradation. Fillers can also be used to add color, opacity, and conductivity to a compound. Unique property combinations can be achieved through the use of fillers [3]. We used Calcium Carbonate as a filler material in the research. The decrease in tensile strength of the PVC products diminishes the tensile-strength relevant friction part and causes decreasing sturdiness. For non plasticized PVC, it is seen that the tensile strength shows a steep increase followed by a rapid decrease and a stable value until the test specimen breaks during the tensile test. That can be seen in the course of the raw material [4].

Each manufacturer optimizes the composition of their product, but in literature there is no information about the optimal composition and effect of combination of different brands of additives on the properties of the product [5]. Various stabilizers are used to give a particular effect to the compound. PVC without any additives, at room temperature, is a rather rigid material. It is often used in place of glass. But if it is heated above the temperature of 87°C a change occurs, PVC becomes flexible and rubbery [6]. Sometimes not all the desired requirements can be attained but formulation with PVC is a good candidate with which to start one's evaluation [7]. The paper outlines the effect of additives mainly plasticizer and filler on tensile strength of polyvinyl chloride compounding. Increasing plasticizer content results in decreasing tensile strength and specific gravity but increasing in elongation at break but they also increase flexibility and extensibility of the product [8-9].

If CaCO₃ filler is properly introduced into the formulation, the desired tensile strength and elastic qualities of the product can be achieved, and it significantly improves the adhesion between the filler particles and the matrix [10-11].

P. Naydenova [8] did the optimization of the composition and technological mode for the production of PVC profiles for doors and windows. He studied the impact of different brands

of stabilizers, modifiers, fillers and pigments on physical and mechanical performance of composites. On the basis of his study, the composition containing stabilizer Chemson GWX523B, FM modifier 50 of company Kaneka, calcium carbonate Hydrocarb 95T and titanium dioxide Kronos 2220 were selected the most suitable.

Quao Jinliang [12] studied the effect of Plasticizers on the microstructure of PS/PVC Blends. When Di-octyl Phthalate (DOP) was used as the plasticizer, the particle size of PVC could not be reduced due to the transference of DOP into PS Phase. When PCL was used, the particle size of PVC could be decreased because PCL does not migrate to PS phase.

Omprakash H. Nautiyal [13] did molding of PVC Air Soles with Modified Formulation. Molding of PVC Air soles was produced with the formulations of Resin (25 Kg), DOP/DBP (8+8 Kg), CPW (3 Kg), R9 (0.750 Kg), Ivamol (0.500 Kg), Stearic acid (0.500 Kg), Foaming ADC (0.350 Kg), MBR (8kg) and Calcium Carbonate. On the basis of the quality evaluations the soles invented with naphthenic oil were found to have superior qualities as compare to the one with CPW and stearic acid.

Safwan Altarazi [14] optimized Materials Cost and Mechanical Properties of PVC Electrical Cable's Insulation by Using Mixture Experimental Design Approach. 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% CaCO₃ with particulate size 10 microns, 14% DOP plasticizer, and 3.5% CPW (Chlorinated Paraffin Wax) plasticizer. For maximum UTS the compound should consist of: 17.5% DOP, 62.5% virgin PVC and 20.0% CaCO₃ of particle size 5 microns. Finally, for the highest ductility the compound should be made of 35% virgin PVC, 20% CaCO₃ of particle size 5 microns, and 45.0% DOP plasticizer.

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) including factorial designs, Taguchi designs, and mixture designs [14]. A factorial design is type of designed experiment that lets you study of the effects that several factors can have on a response. When conducting an experiment, varying the levels of all factors at the same time instead of one at a time lets you study the interactions between the factors

2. EXPERIMENTATION AND PREPARATION

Compounds were made by measuring the weight of each of the components in a digital balance and all of the components were mixed by mechanical mixing so that a good homogeneous mixture is prepared.

2.1 Material

PVC Resins (Ethyne-octene copolymer) having K- Value of 67 was used. They were bought from LG Chem. Test samples with filler were prepared with the PVC and additives such as stabilizer, lubricant and plasticizer. The particle size of PVC Resin was of 99.8 microns. Two plasticizers were used namely, di-octyl phthalate (DOP) and Chlorinated Paraffin Wax (CPW). DOP C8 with specific gravity value of 0.9 is used as a primary plasticizer. Chlorinated Paraffin Wax (CPW) is used as an optional secondary plasticizer. Here the CPW used was of type Grade II CP 52 which contains about 52 % of Chlorine. Calcium carbonate (CaCO₃) is used as important reinforcing filler in thermoplastic industry and has been studied by many researchers who have reported large improvements in mechanical properties such as strength, modulus and toughness. The uncoated precipitated Calcium Carbonate is used in the process having size of about 3.5 microns. All of the samples contain 2-3% of Stabilizers with main constituents as Tri Basic Lead Sulphate (TBLS) Epoxidized Soyabean Oil (ESO), stearic acid and colorants.

2.2 Compounding



Fig. 1: Batch Mixing Extrusion Machine

For all the composite formulations weight of resin, stabilizer, lubricant and plasticizers were varied and Calcium Carbonate was added as filler to this mix in different weight proportions. The research work was aimed to determine use of Calcium Carbonate as filler and the role of composition of PVC Resins and plasticizers in PVC to develop composite with improved mechanical properties. The weight proportions of the PVC Resin, Calcium Carbonate (CaCO₃), Di-octyl Phthalate (DOP) and Chlorinated Paraffin Wax (CPW) were selected based on the PVC cable formulations which is given in Table 1.

Table 2: Upper and Lower Limits of the components

Component	Lower Limit	Upper Limit
PVC Resin	40	50
Calcium Carbonate (CaCO ₃)	19.34	29.82

Di-octyl Phthalate (DOP)	8.70	12.04
Chlorinated Paraffin Wax (CPW)	9.65	19.44

Various blends of PVC were prepared with the help of Batch mixture Machine with which the extrusion process was done. Varying composition of PVC Resin was used in each case. The batch mixtures were produced having total weight percentage of the plasticizer was limited to 19.55% - 31.48%. After three different heating zones were provided for heating up the PVC having temperatures of 130°, 140° and 160°, after which it was cooled in the water bath. Dry blended PVC mix was compounded with the help of 60 r.p.m. roller speed for all batches. Nine batches of such compound were processed with this machine for and each formulation of composite contained different compositions for all of the mixing components. Processed PVC mix was found non sticky and was able to remove from blades easily. All the samples contained stabilizers for about 2-4% by weight to impart required physical and mechanical properties to the PVC.

2.3 Ultimate Tensile Testing



Fig. 2: Samples made from PVC

Nine samples were made from each of the mixture and they were made according to IS: 10810 (Part 7) with inner diameter and outer diameter were taken as 1.5 mm and 2.7 mm respectively and the marker lines were set to 2 mm. The testing was done on Universal Tensile Strength Machine (Kamal Metal Industries). The Ultimate Tensile Strength for each of the material was found out. The following table shows the various results taken out in the Testing process.

Table 2: Ultimate Tensile Results for different Mixtures

Mixture	Resin	CaCO3	CPW	DOP	UTS
X1	43.86	29.82	11.40	9.65	13
X2	50.00	20.00	12.00	10.00	16
X3	47.89	23.95	10.92	13.41	13.5
X4	46.99	23.5	10.71	15.04	11.8
X5	48.36	19.34	11.03	15.47	15.3
X6	46.70	25.42	10.42	12.80	13.8
X7	45.45	27.27	10.91	12.73	15
X8	40.04	27.78	12.04	19.44	13
X9	42.48	28.26	8.70	10.87	12

3. RESULTS AND DISCUSSIONS

Minitab was used to analyze the results been made up in the experimentation. The analysis is conducted in three stages: Analysis of variance (ANOVA), Surface Plots, Scatterplot Graphs and Response Optimizer.

The next subsections describe the analysis details.

3.1 Analysis of Variance (ANOVA)

Table 3: ANOVA Table for UTS

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	6	10.474	3.4913	45.2	0.109
Resin	1	4.22	10.212	10.282	133.13	0.055
CaCO3	1	1.77	7.1765	7.1765	92.92	0.066
P (Plasticizers)	1	0	5.9466	5.9466	76.99	0.072
2 Way	3	4.19	11.1213	3.7071	48	0.106
Resin*CaCO3	1	3.96	7.5085	7.5085	97.22	0.064
Resin*P	1	0.02	5.3344	5.3344	69.07	0.076
CaCO3*P	1	0.2	6.2829	6.2829	81.35	0.07
3 Way	1	7.38	7.3876	7.3876	95.65	0.065
Resin*CaCO3*P	1	7.38	7.3876	7.3876	95.65	0.065
Residual Error	1	0.07	0.0772	0.0772		
Total	8	17.66				

Setting type one error (α) at 0.05, any effect with a P-value less than α indicates significance. Table 3 shows the results made in ANOVA. The 2 way and 3 way interactions is seen in Table 3. None of the P-values were found to be under the value of α .

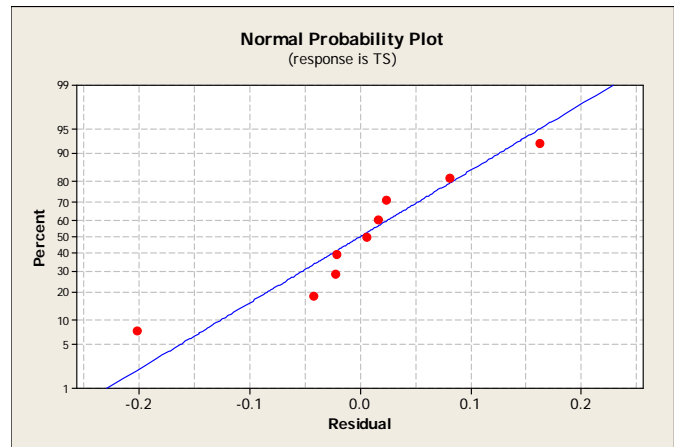


Fig. 3: Normal Probability Graph

The normal probability plot is shown in Fig. 1. The normal probability plot of the residuals and the histogram do not indicate any deviation from normality, thus, normality assumption is appropriate. No values under 0.05 were seen so there is no significant interaction between any of the components.

3.2 Surface Plots

Mid settings were chosen to see the effect of various components in the Tensile Strength. In Fig. 4, the Surface plot between CaCO_3 and DOP for Tensile Strength, we get a plane surface plot. The Tensile Strength is at maximum on the upper left of the diagram where Calcium Carbonate is about 20% and DOP is near to 10%-15%. Saddle point is shown in Fig. 3 in which we get the plot between PVC Resin and CaCO_3 . The graph shows the points, a point in the domain of a function that is a stationary point but not a local extremum.

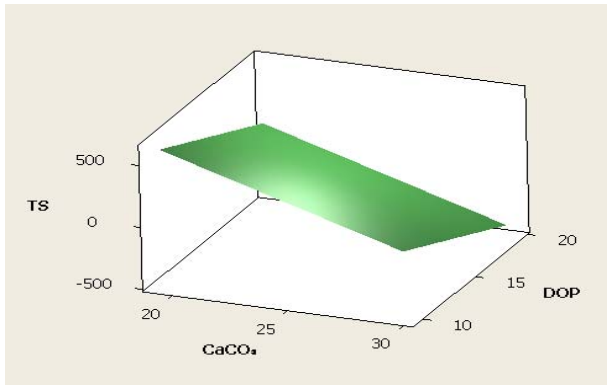


Fig. 4: Surface plot between CaCO_3 and DOP for Tensile Strength

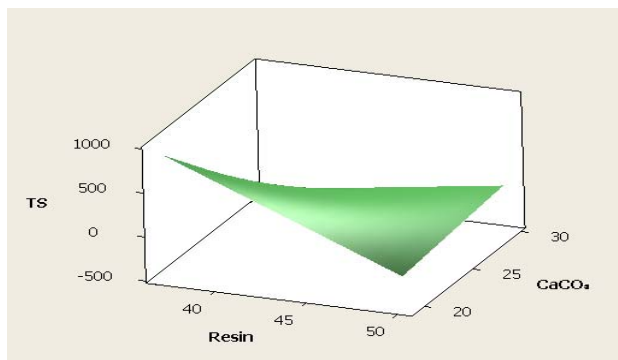


Fig. 5: Surface plot between PVC Resin and CaCO_3 for Tensile Strength

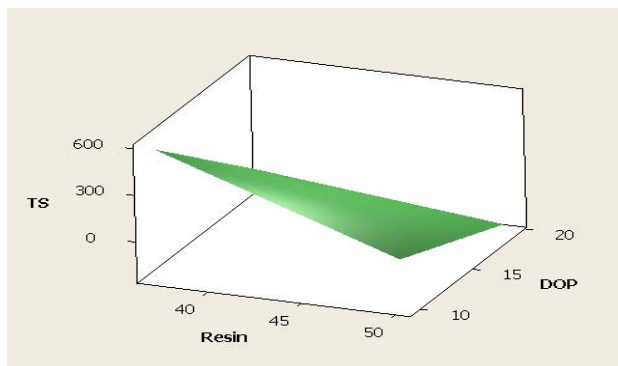


Fig. 6: Surface plot between PVC Resin and DOP for Tensile Strength

Fig. 6 represents the plot between PVC Resin and DOP for Tensile Strength showing great variation in Tensile strength. All the graphs show that the Calcium Carbonate decreases the Tensile Strength and DOP and PVC Resin increases the Tensile strength of PVC.

3.3 Scatterplot Graph

The variation of Ultimate Tensile Strength with the change in composition of the constituent materials can be showed with the help of Scatterplot Graphs for each of them. The change in Ultimate Tensile Strength occurs due to change in the composition of PVC, and its value is totally a result of the way in which the compounding is done. Each of the mixture has mechanical properties as a direct contribution of the percentage of each constituent material. Scatterplot graphs with regression and groups were made. The cubic model was followed which gave the results as shown in the following figures.

Fig. 7 represents the relation between the changes in UTS with the change in the change in the percent composition of PVC Resin. Ultimate Tensile Strength is continuously increasing for PVC Resin for the range 40% to 50%.

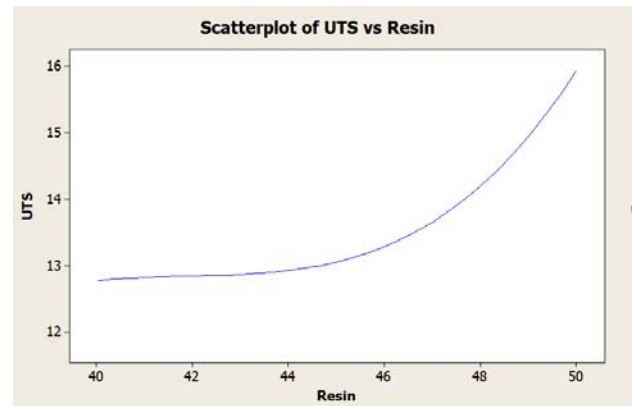


Fig. 7: Scatterplot Graph between UTS and PVC Resin

In Fig. 8 the UTS is continuously decreasing with increase in the percentage of DOP in the compounding. The variation in UTS is not very significant but it can be clearly seen that it is decreasing with the increase in the composition of DOP.

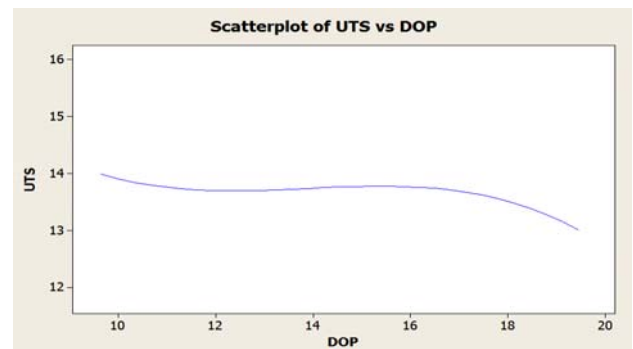


Fig. 8: Scatterplot Graph between UTS and DOP

Fig. 9 shows the relationship between the UTS and the composition of CPW in the compounding. Though, CPW is added to enhance the properties of DOP and it is doing the same here also. The increase in the percentage in DOP decreases the UTS, in other hand, CPW is increasing UTS. Calcium Carbonate, being a filler material decreases the UTS of PVC as seen in the Fig. 10.

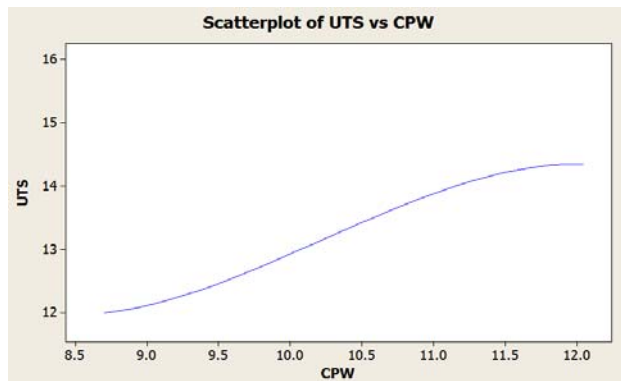


Fig. 9: Scatterplot Graph between UTS and CPW

There is a bend in the curve in Fig. 10 that must be due to the interactive chemical properties of the constituent materials. The UTS tends to increase for a very little range between 24% to 27% after which the graph shows a steep downward slope.

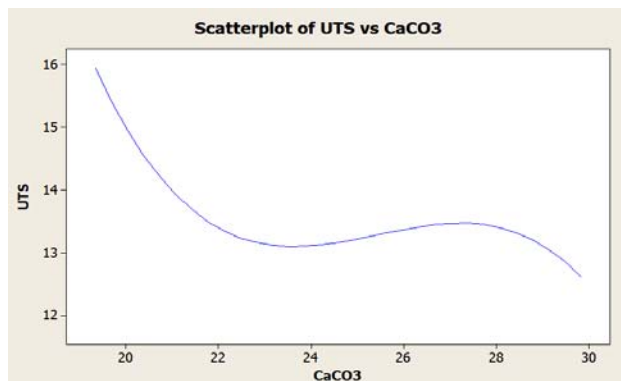


Fig. 10: Scatterplot Graph between UTS and CaCO₃

3.4 Response Optimizer

The response optimizer in Minitab was used to determine the optimal components percentages that will produce the best output/response values for the UTS. The response optimizer model was built with the purpose of Targeting UTS. The Goal was targeted to 12.5 with lower and upper limits set to 11 and 14 respectively. The importance was kept at level 2. The Results of the Response Optimizer is shown in the Table 4.

4. CONCLUSION

The Ultimate Tensile Strength increases with the increase in the content of PVC Resin and CPW, whereas it decreases with the increase in the composition of DOP and CaCO₃. The normal probability plot of the residuals and the histogram do

not indicate any deviation from normality, thus, normality assumption is appropriate. The maximum Ultimate Tensile Strength of 15.3 N/mm² was observed for the sample X5 that contains 48.36% PVC Resin, 19.34% CaCO₃, 11.03% CPW and 15.47% DOP. On Optimizing the Ultimate Tensile Strength as per the standards of IS: 5831–1984 with the help of Response optimizer, the results are as shown:

Table 4: Optimized Results of Response Optimizer

PVC Resin	CaCO ₃	CPW	DOP
49.58%	29.18%	10.90%	9.65%

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