Synthesis and Applications of Non-woven Polyester Fabric-Reclaim Butyl Rubber Composite

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***Abstract* - Non-woven cross-linked polyester fabric cutting waste from an apparel industry has been recycled through mechanical recycling route to form a novel composite material with Reclaim Butyl Rubber (RBR). Fourier Transform Infrared Spectroscopy (FTIR) spectra of the polyester fabric revealed the cross-linked nature of the material with the presence of multiple functional entities. The fabric is found to undergo thermal degradation in the range of 240-300°C, indicated by the peaks of Differential Scanning Calorimetry (DSC) curve. This study focuses on utilizing the strength and durability of the two raw materials involved to form a Polyester Reinforced Rubber (PRR) with improved stability and process ability. Introduction of plasticizer at the compounding stage has imparted exceptional surface finish to the cured composite. A detailed chemical and mechanical analysis of PRR has been done, which indicates the compatibility of the two polymers and enhancement in the hardness of the reclaim butyl rubber by around 10-15 units. The PRR has then been subjected to compression molding to fabricate commercial products like insulating films and gaskets, which exhibit significant electrical insulating properties and chemical stability. A brief economic analysis of PRR gasket and virgin butyl rubber gasket shows a reduced cost margin of 14.6% in the processing cost of the former, validating the market potential of the product. The utilization of these industrial waste materials, whose current disposal practices pose a concern at environmental level, into serviceable products gives a route for sustainable recycling practices.**

***Index Terms* - Compression molding, Gasket, Insulating material, Polyester reinforced rubber, Recycling, Sustainability.**

I.INTRODUCTION

Polyester has emerged as a popular fabric material over its natural counterpart, owing to its unique set of properties which include versatility and recyclability. Textile industry has been witnessing a notable shift in momentum in terms of consumption pattern from cotton to synthetic fibres over past few decades. Polyester has a dominant share of fibre consumption, accounting 36% of global market share, as marked from the Global Nonwovens Staple Fibre Consumption report 2018 [Fig.1]. It is being expected that till 2030, it will occupy 60% of total fibre demand on global level [1].The consumption of polyester fibre in India has shown an exponential rise with just 139 kg per year, in 1992 to 713 kg per year, in 2018 [2]. Polyester has continued taking up the share as it provides an economical option to fulfil the demand gap, with faster processing time and requirement of lesser amount of water during the manufacturing stage.

Polyester fabric is effectively utilized in a wide range of applications: in apparel as textile fabricated products due to its tenacity and durability; in home furnishing applications such as carpets and mats, owing to its rigidity; and its insulating properties make it suitable as industrial textile. This increased levels of consumption in several applications has resulted in polyester fabric gaining a control over the clothing industry, with a yearly output of more than 22.67 billion tones worldwide [3]. It is evident from the above-mentioned figures that there is a considerable amount of post-industrial as well as post-consumer fabric waste being generated. Therefore, in the light of environmental consequences and economic issues, there is a growing interest in the field of recycling the fabric waste to produce valuable products.

The current methods of disposal of polyester fabric waste namely landfills, open-air burning and incineration are not efficient as they result in great wastage of valuable materials [4]. Moreover, the environment where these materials are dumped is affected adversely and is raising serious concerns. Therefore, it is imperative to find more effective methods to treat polyester fabric waste.

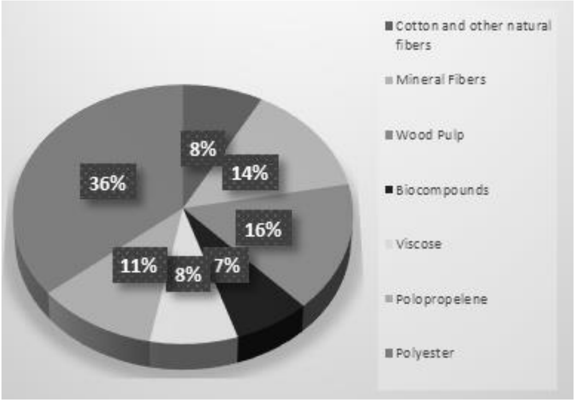


Fig.1 Global non-woven fiber consumption, 2018 [1]

Considering the diversity of fibrous waste and structures, many technologies must work with an integrated approach in order to have a considerable impact on waste recycle. Polyester fabric waste normally occurs in composite form, containing blends of pure fibres, reinforcements and fillers to enhance several properties depending on the end use. Since each component in this composite would have different properties, employing a general recycling method to treat all sorts of composites is not feasible. Polyester fabrics are well known for their superior properties such as mechanical strength, thermal stability, toughness, flame retardancy and chemical stability, which makes them applicable for a wide range of applications. However, recycling such materials is often difficult due to their overall structural stability. Another barrier in recycling of such waste is lack of equipment and technology and unwillingness of manufacturers to participate in development and production of serviceable products from waste fabric. To the recycler, waste is not viewed as a garbage, but rather is valued as a commodity [5]. There has been a considerable increase in recycling of fabric waste in the past two decades. In spite of several challenges in the way of recycling, the tide of fabric waste headed to conventional solid waste disposal is slowly being turned. The various possible routes through which non-woven polyester fabric waste can be recycled are broadly classified as mechanical, thermal and chemical [6].

The polyester fabric can be designated as a reinforcing filler whose primary function is to improve the mechanical properties of the rubber compound, the fibre-based composites may sometimes also impart some adequate functional properties to the product. Lawandy et al formed butyl rubber coated polyester fabric with various concentrations of MT carbon black in the rubber. It was observed that the dielectric properties of rubber-fabric assembly correlated with peel strength [7]. In another such research, Basim Abu-Jdayil et al determined the mechanical behaviour of rubber unsaturated polyester composites, to be used as insulating material. The experimental results revealed that mixing rubber particles with the polyester matrix decreased both compressive and tensile strength of the composite, however it was still high as compared with commercially available insulating material [8]. Halim et al presented a new method for improving adhesion between natural rubber and polyester fabric. This was successfully done by in situ grafting of maleic anhydride onto natural rubber in a two-roll mill. The grafting procedure was followed by infrared spectroscopy [9]. Another similar research carried out by A.A. El-Wakil presented a method for improving adhesion between ethylene propylene diene monomer (EPDM) rubber and polyester fabric. In this work, natural rubber was modified by maleic anhydride in order to improve the adhesion force between EPDM rubber and polyester fabric [10]. Mente P. reviewed the tensile, curing, viscoelastic, morphology and thermal properties of natural fibre reinforced rubber composites and reclaimed rubber. Overall reinforcement of rubber has shown to improve properties like modulus; tensile strength; tear strength and elongation [11].

Recycling the polyester waste has been studied over many years by the researchers for application in various fields including civil structures, energy recovery and chemical blends. However, there are very few areas in which research has been done to exploit the waste polyester non-woven fabric which is highly cross-linked.

This work focuses on the mechanical recycling route which involves physical reprocessing of the fabric by employing basic unit operations such as blending, compounding and moulding with reclaim butyl rubber to form serviceable products. The mechanical recycling should not bring any change in the chemical structure or properties of the polymers involved. As compared to other routes, mechanical reprocessing is relatively simple, requires low investment, does not require sophisticated equipment, energy demand is low and most importantly has negligible environmental impact. Thus, it shows a great potential for low-cost start-ups to generate local employment as well as reutilization of waste fabric. Also, considering the complex chemical structure of the cross-linked polyester [Table III] it is challenging to come up with an exact chemical mechanism to bring about its chemical or thermal recycling. This has been confirmed through a series of experiments of unsuccessful dissolution of the fabric in many solvents as well as combustion experiments which generated no valuable product but toxic fumes.

II. MATERIALS AND METHODS

A. Materials

The non-woven polyester fabric used in this work was received from Helsa Icon India Pvt. Ltd., Hyderabad, in the form of strips of irregular dimensions. Reclaim butyl rubber and the industrial grade sulphur, MBT, TMTD and Z-150 compounding agents were kindly provided by GRP Ltd., Ankleshwar plant.

B. Fourier Transform Infrared Spectroscopy (FTIR)

The fabric sample was analysed at the lab of UPL Ltd. Ankleshwar, with PERKIN ELMER Spectrum-2 model to obtain the FTIR spectra in order to determine the functional groups present in the polymer to find its most probable chemical composition. The FTIR spectra was obtained in terms of % Transmission and wavelength, in the range 8300-350 cm^{-1}.

C. Melt Flow Index

Melt flow index was measured in a PRESTO melt flow tester 220V, single phase 50 Hz with a temperature range of 400℃ and resolution of 0.1 ℃. This index helps to investigate the melt and flow properties of the polymer. It is calculated in terms of the amount of material measured in grams that exits the die in 10 minutes.

D. Differential Scanning Calorimetry (DSC)

DSC analysis of sample was conducted at the lab of UPL Ltd, Ankleshwar in STARe SW 15.00 grade apparatus. The temperature range for the analysis was taken ambient to 400℃, at a heating rate of 5℃/min.

E. Ultimate and Proximate Analyses

The fabric sample was subjected to ultimate and proximate analyses conducted at BEIL Ltd., Ankleshwar in order to determine its organic composition and calorific value. The % Loss on drying was determined by heating the sample at 110℃ for 2 hours in a hot air oven. Ash % was calculated by placing the sample in a muffle furnace at 800℃ for 2 hours. The elemental analysis of the sample was carried out in a TruSpec CHNS LECO instrument. The calorific value was determined in a bomb calorimeter with an operating range of 200-10000 cal/g.

F. Method

The preparation of PRR samples was conducted at GRP Ltd., Ankleshwar. The reclaim butyl rubber (RBR) was manufactured by thermochemical de-vulcanization of the scrap tyre tubes. This recycled rubber when incorporated in the recipe of chemicals provides faster mixing & extrusion, and also reduces power consumption.

Table I. List of Compounding Agents

CHEMICALS	FUNCTION
Sulfur	Vulcanization Agent
MBT (2-mercaptobenzothiazole)	Primary Accelerant
TMTD (tetramathylthiuram disulfide)	Secondary Accelerant
Z-150	Plasticizer

The polyester fabric was introduced during the compounding process carried out on a two-roll mill with a roll diameter of 200 mm and the roll speed range of 1.6-15 m/min, during which the specific combination of chemicals [Table I] is added to the rubber, in order to enhance the curing mechanism.

Seven samples were prepared weighing 200 g each and varying the mass percentage of fabric and RBR in each sample by keeping the amount of plasticizer constant [Table II]. This was done to study the effect of addition of fabric on qualitative parameters of the rubber.

The qualitative testing of the compounded composite samples was carried out as per IS 6306 to calculate the chemical and processing parameters. The compounded samples were then subjected to Rheological analysis in order to determine the variation of curing condition with changing composition in the samples and also to check the effect of plasticizer on the curing time. The compounding process was followed by curing the formed mass of rubber under compression molding for 30 minutes at

160℃. Curing was carried out in moulds whose shape and size can be modified depending on the specifications of rubber product which is intended to be manufactured. Cured PRR was then subjected to mechanical parameter testing [IS 6306].

G. Product Application and Testing

After conducting the preliminary trials it was concluded that better surface finish and mechanical properties are encountered in sample SBZ-6. Thus, considering it to be the optimum composition, the compound was then moulded into two industrial products: electrical insulation film and gasket.

Table II. Composition of Samples

Sample	Quantity of RBR (g)	Quantity of Polyester (g)	Quantity of Plastizer (g)
SBZ-0	200	0	5
SBZ-1	195	5	5
SBZ-2	190	10	5
SBZ-3	180	20	5
SBZ-4	170	30	5
SBZ-5	160	40	5
SBZ-6	150	50	5

Electrical Insulation test in a Sphere Gap Assemble (30kV and 30 mA capacity) manufactured by Moon light Electricals, was performed in Electrical Department at SRICT. Sample was placed on the metal plate and the voltage plug was placed on the sample thus forming a closed loop circuit. The supplied voltage is progressively increased and the value of breakdown voltage of the sample was reported on the panel when the material gets ruptured. Dividing the breakdown voltage by the thickness of the sample gives its dielectric strength.

The SBZ-6 compound was cured in a gasket mold, at 160 ℃ for 30 minutes, with capacity of 5 gaskets per batch. The standard test to examine the applicability of the gasket in a chemical environment is done by carrying out swelling test (ASTM D3616). Gasket was simply immersed in solvents like water, methanol, phenol and other petroleum fractions for a course of 3 and 5 days and its weight before and after the process was recorded. This is followed by determination of % swelling by the following formula [13]:

$$\% \text{ Swelling} = \frac{(X - X_0) * 100}{X_0}$$

III. RESULTS AND DISCUSSION

A. Material Characterization

The FTIR spectra of sample [Fig. 2] was studied thoroughly and the functional group interpretation derived from the values of wavelength [14] is as shown in Table III. The peak obtained at the wavelength of 1713.22 cm^{-1} indicates the presence of unsaturated ester group and that at 722.39 cm^{-1} defines aromatic stretching vibration in benzene ring, thus confirming the structural composition of aromatic ester. However, the spectral peaks at various other wavelengths indicate that the fabric is a highly cross-linked material containing functional groups mentioned in Table III.

In case of melt flow index analysis of the polyester fabric sample it was observed that the material did not melt, however it started to release fumes at undergoing complete thermal decomposition without forming any melt. Therefore, the test confirmed that the fabric cannot be converted into liquid phase by melting.

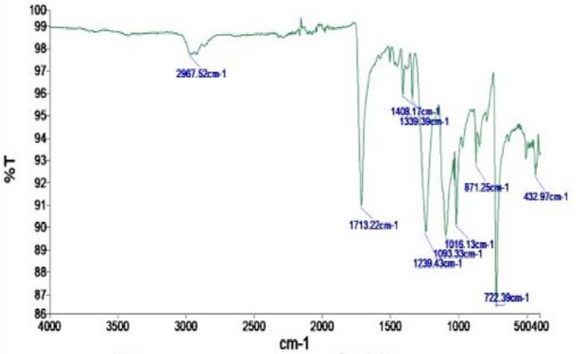


Fig.2 FTIR spectra of fabric

Table III. Functional group interpretation of spectra

Peak No.	X (cm^{-1})	Y (%T)	Range	Functional Group
1	2967.52	97.76	2950-2970	C-H Stretching
2	1713.22	90.99	1715-1730	Unsaturated esters
3	1408.17	95.95	1310-1410	O-H Bending
4	1339.39	95.78	1340-1365	Sulfonates
5	1239.43	89.80	1230-1270	Aromatic ethers
6	1093.33	89.63	1094	C-O-C Streatching
7	1016.13	90.15	1020-1070	C-O Streatching
8	871.25	92.87	820-890	C-O-O Streatching
9	722.39	86.47	720-750	Benzene ring
10	432.97	92.38	470-500	Polysulfides

The presence of two endothermic peaks in the DSC curve [Fig 3] indicate that two different components are thermally decomposing at different temperatures [Table IV]. The results of DSC analysis agree with the observation of melt flow index test, as the temperature range for thermal decomposition is within the same limits.

Table IV. Result of DSC analysis

ENDOTHERM	LEFT LIMIT	MELTING POINT	RIGHT LIMIT
PEAK-1	240.59 °C	244.48 °C	247.01 °C
PEAK-2	252.25 °C	254.54 °C	258.80 °C

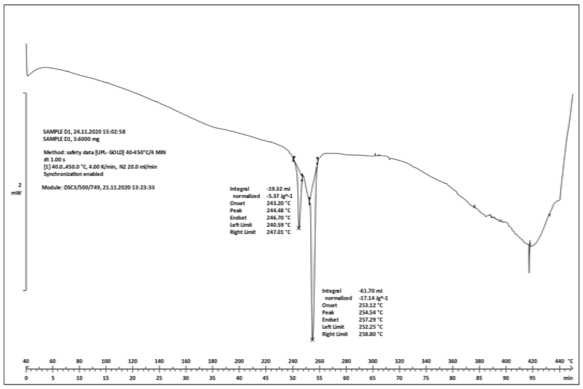


Fig. 3 DSC curve of fabric

The results of ultimate and proximate analyses highlights the high volatile matter content of above 95% and significant calorific value of 5954 cal/g.

Table V. Ultimate and Proximate Analysis Results

Parameters	Value
Loss on Drying	0.194%
Ash Content	0.39%
Volatile Matter	95.91%
Fixed Carbon	3.51%
Carbon	60.2%
Hydrogen	4.29%
Nitrogen	0.592%
Sulphur	0.06%
Calorific Value	5954 cal/g

B. PRR Qualitative Analysis and Composition Optimization

a. Electrical Insulating Material

The results of Sphere gas assembly test have shown an increasing trend in the dielectric strength of the samples with increase in the fabric composition, the highest being noted for the sample SBZ-6 [Fig. 6], which even displayed satisfactory results of mechanical properties.

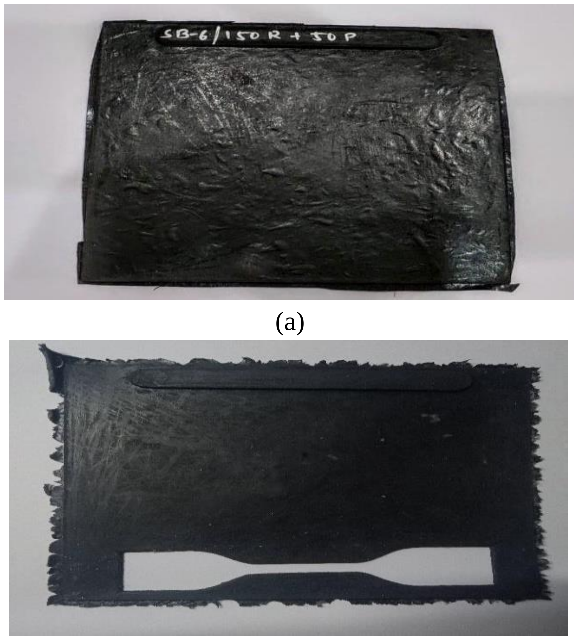
b. Gasket

Swelling test was carried out with organic solvents where gaskets are likely to be used. Phenol, Methanol and steam gave positive results with the composite material, with % swelling less than 25% [Table X]. Thus the gasket can be used in the equipment where the solvents are under function.


(a) (b)

Fig.4 PRR Moulded Products (a) Insulation Film (b) Gasket

Another parameter which is tested at the industrial level for gasket manufacturing is its hardness. The standard range of hardness for butyl rubber is found to be in the range of 60-70 shoreA [12] [15]. The hardness of SBZ6 composite (Table IX) is 61 shoreA, which is well within the standard range.



(a)

(b)

Fig.5 Cured Samples before (a) and after (b) the addition of Plastizer

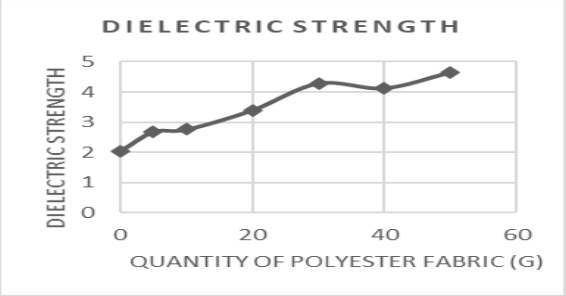


Fig.6 Variation in Dielectric Strength with Fabric Composition

Table VI. Chemical Parameters of PRR Samples

Sample	*%Ash*	*%Carbon*	*Acetone Extract*	*% Volatile Matter*	*% Rubber Hydrocarbon*
SBZ-0	5.54	30.72	8.17	0.19	55.38
SBZ-1	5.75	32.04	8.36	0.22	53.63
SBZ-2	5.82	31.38	10.4	0.25	52.15
SBZ-3	5.86	30.33	10.55	0.19	53.07
SBZ-4	5.58	32.24	10.81	0.19	51.18
SBZ-5	4.27	33.22	10.64	0.19	51.68
SBZ-6	4.22	31.52	10.63	0.2	53.43

Table VII. Processing Parameters of PRR Samples

Sample	*Mooney Viscosity*	*Specific Gravity*
SBZ-0	32.01	1.14
SBZ-1	36.49	1.14
SBZ-2	37.7	1.13
SBZ-3	44.2	1.14
SBZ-4	49.84	1.12
SBZ-5	50.88	1.13
SBZ-6	55.26	1.13

Table VIII. Results of Rheological Analysis

Sample	*Tc90 (min)*
SBZ-0	22.29
SBZ-1	24.52
SBZ-2	25.18
SBZ-3	20.29
SBZ-4	25.21
SBZ-5	29.04
SBZ-6	27.09

Table IX. Mechanical Properties of PRR Samples

Sample	*Hardness (Shore A)*	*Tensile Strength (kg/cm^2)*	*%Elongation*
SBZ-0	52	41.95	540
SBZ-1	68	29.45	410
SBZ-2	74	28.14	400
SBZ-3	62	20.3	280
SBZ-4	68	22.74	310
SBZ-5	61	25.24	340
SBZ-6	61	33.92	440

Table X. Results of Swelling Test

Solvent	*X_0 (g)*	*X (g)*		*%Swelling*	
		3 days	5 days	3 days	5 days
Toluene	2.946	4.812	4.842	63.34	64.35
Petrol	3.15	4.925	4.985	56.35	58.25
Kerosene	3.717	5.718	5.742	53.83	54.47
Diesel	3.827	5.405	5.411	41.23	41.39
Methanol	2.915	3.067	3.098	5.21	6.27
Water[1]	2.24	2.412	-	7.67	-
Phenol	2.273	2.495	-	9.766	-

Swelling Test was carried out at 100°C in a water bath.

C. Cost Analysis of PRR Gasket

There is found to be an overall reduction in the processing cost, with a margin of 14.6 % for RBR-Fabric gasket over its virgin rubber counterpart [Table XI]. The cost reduction has been attributed by the fact that per unit cost of RBR is less than that of virgin butyl rubber. Also, fillers such as carbon black and processing oil are not required in case of RBR-fabric gasket, which further results in decrease in cost per gasket.

IV.CONCLUSION

On working with the objective of providing an environmentally viable and economic solution to the industry for reutilizing the polyester non-woven fabric waste, the mechanical recycling by compounding the fabric with reclaim butyl rubber resulted in a composite, having chemical compatibility between the two polymers. Preliminary trials were conducted to determine the optimum composition of fabric and RBR in the composite as well as the effective combination of curators and activators for the curing process. The fabric composition of 25%, with incorporation of 5g of plasticizer formed a composite with acceptable range of mechanical properties and imparted exceptional surface finish to the cured material. This compounded sample was then molded to give electrical insulating slabs with a breakdown voltage of 12kV and consequent dielectric strength value 4.63 kV/mm. With the same compound ratio, the sample was molded into gaskets, resulting in the hardness in the range of 60-65, which is an acceptable range as per the industrial standards for butyl rubber material. Also the swelling test with % swelling of less than 25% in each of these solvents. The cost analysis reveals a reduced margin of 14.6% in the gaskets produced from the polyester fabric-RBR composite as compared to that of virgin butyl rubber. With these outcomes, there is a scope of further research in the direction of mechanically blending the fabric with other grades of rubber and another set of polymeric materials to form novel composites.

Table XI. Cost Analysis of Virgin Butyl Rubber Gasket and PRR Gasket

Material	*Unit*	*Rate (Rs)*	*Virgin Rubber Gasket*		*Reclaim Rubber Gasket*	
			Quantity	Cost	Quantity	Cost
Butyl Rubber	kg	180	0.200	36.000	0	0
Reclaim Butyl	kg	80 [16]	0.000	0.000	0.15	12
Polyester fabric	kg	5	0.000	0.000	0.05	0.25
Oil	kg	42[17]	0.005	0.210	0	0
Carbon Black	kg	55	0.030	1.650	0	0
Z-150	kg	50	0.005	0.250	0.005	0.25
Sulfur	kg	26[18]	0.002	0.052	0.002	0.052
MBT	kg	320[18]	0.001	0.160	0.0005	0.16
TMTD	kg	195[18]	0.001	0.195	0.001	0.195
Zinc Oxide	kg	190[18]	0.005	0.950	0.005	0.95
St.Acid	kg	88[18]	0.000	0.000	0	0
SubTotal		1231	0.2485	39.467	0.2135	13.857
Power				36		36
Manpower Cost				100		100
Total For 5 Gasket				175.467		149.857
Per Gasket rate				35.0934		29.9714
Reduced Cost Margin Over Virgin Rubber Gasket (%)						14.595

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