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Novel dual peptisers and curing agents derived from PET waste as *N*-alkyl benzene dicarboxamides for EDPM waste

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Ethylene-propylene-diene monomer (EPDM) is widely used in the manufacturing of various components in rubber industries and hence recycling of scrap rubber is a major problem. In the present study, EPDM rubber waste powder has been recycled successfully by using *N*-alkyl benzene dicarboxamides, derived from PET wasteand other additives. Three aminolysed end products namely 1,4-benzene dicarboxamide, 1,4-benzene dicarbohydrazide and *N*,*N*′-diaminoethyl 1,4-benzene dicarboxamidewere used as peptisers and curing agents in the EPDM waste formulations. EPDM rubber waste sheets have been prepared at comparatively low temperature using two roll mill and compression moulding machine. Tensile strength, % elongation and hardness have been found in the range 12.23-20.19 kg/cm^2, 107-145 % and 46-61, respectively. It has been expected that the developed reclaimed process described in this paper will promisingly support the protection of environment and conservation of resources with favors to rubber waste generated throughout the world.

Keywords: EPDM, Recycling, Curing, PET waste

1 Introduction

Rubber is one of the most versatile materials for different sectors including healthcare, automotive, chemical manufacturing, refining, food processing, electronics, semiconductor manufacturing and pharmaceutical industries etc. Because of its unique and remarkable properties such as capable to dampen or reduce vibrations, high resistance to alkali, acids and chemicals, it possess wide spectrum of applications. Ethylene-propylene-diene monomer (EPDM) is a thermoplastic elastomer which is widely used in the manufacturing of various components in rubber industries and hence recycling of scrap rubber is a mainconcern[1-3]. Recycling of waste and development of applications for recycled products is the current state of art[4]. The reported literature details the incorporation of waste rubber into numerous types of matrix such as concrete, latex, thermoplastic and thermoset materials[5-11]. Very few reports are available regarding recycling of EPDM rubber waste because there are so many problems related with recycling of EPDM rubber such as higher percentage of stable monosulfidic crosslinks and less solubility of most devulcanizing agents[12-17]. New methods for recycling of waste have always been in demand for protection of environment and provide a way to reuse the recycled products[18].

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Herein, an attempt has been made to recycle EPDM rubber waste using chemically recycled derivatives of poly-ethylene terephthalate waste 1, 4 benzene dicarboxamide, 1, 4 benzene dicarbohydrazide and *N*,*N*′-diaminoethyl 1,4-Benzene dicarboxamide with other additives; stearic acid, sulphur, ZnO and naphthalene oil by using two roll mill and compression molding pressure machine. The sheets were analyzed for mechanical properties such as tensile strength, % elongation and hardness. Effect of different amides on Mooney viscosity, cure and mechanical performance of EPDM waste rubber has been investigated.

2 Materials and Methods

The EPDM rubber waste used for this study was procured from Partapur, Meerut. Liquor ammonia (25% w/w), hydrazine hydrate (99%) and ethylenediamine (99%) were procured from Fisher scientific. Post-consumer PET bottles were used for the synthesis of different amides such as 1,4-benzene dicarboxamide, 1,4-benzene dicarbohydrazide and *N*,*N*′-diaminoethyl 1,4-benzene dicarboxamide. Naphthalene oil (99%) was procured from SDFCL, stearic acid (90%) and zinc oxide (99%) were of Qualigens and sulphur (98%) was procured from CDH[9,10,19].

2.1 Mixing

The compounding of different ingredient with EPDM rubber waste was performed at room temperature using

a high speed mixer. The composition of the formulations are given in Table 1 with varying amounts (1, 1.25, 1.5 and 2 g) of 1,4-benzene dicarboxamide (TP1-TP4), 1,4-benzene dicarbohydrazide (TDH1-TDH4) and *N*,*N*′-diaminoethyl 1,4-benzene dicarboxamide (BAETP1-BAETP4).

2.2 Preparation of reclaimed EPDM rubber sheets

The waste EPDM rubber powder was mixed with 1,4-benzene dicarboxamides and other additives such as naphthalene oil, zinc oxide, stearic acid and sulphur using high speed mixer until the free flowing powder is obtained. The powder was processed in two roll mill (maintained at temperature 70 °C) until a uniform sheet was obtained. Mooney viscosity and rheometry experiments of the compounded rubber were conducted to evaluate the scorch time and curing parameters.The compounded rubber was moulded into sheet using compression moulding machine (maintained at temperature 160 °C and pressure 40 kg). A smooth and void free EPDM sheet was obtained. The sheets thus obtained were analysed for various mechanical properties such as tensile strength, percentage elongation and Hardness.

2.3 Methods of testing

2.3.1 Mooney viscosity and scorch time

Mooney viscometer (Microvision Enterprises, Sonipat, Haryana) at 120 °C as per standard ASTM D1646-96a was used for the determination of Mooney viscosity for the compounded reclaimed EPDM waste rubber. The scorch time was taken for a rise of 5 units from the minimum torque at that temperature.

2.3.2 Rheometric characteristics

M_L (minimum torque), M_H (maximum torque) and T_{c90} (optimum cure time), were determined using an Oscillating disc rheometer (ODR) (Microvision Enterprises, Sonipat, Haryana). The measurements were carried out at 160 ± 0.5 °C.

2.4 Determination of mechanical properties

Sheets with thickness of 1.5 mm were prepared for each formulation and used to determine tensile strength, % elongation and hardness. The hardness of sheets was determined using a Shore-A durometer as per standard ASTM D2240. The tensile properties of the casted reclaimed EPDM rubber sheets were determined (ASTM D 638) with the help of Universal Testing Machine (Fine Spavy associates). Tensile strength was determined keeping straightening speed of 100 mm min^{-1} at a temperature of 28 °C. The values of tensile strength and % elongation at break were calculated by following equations:

Tensile Strength = Value of load / Thickness × Width

% Elongation at break = (Final length – Initial length / Initial length) × 100

3 Results and Discussion

3.1 Mooney viscosity and scorch time

The data summarized in Table 2-4 shows the continuous reduction in Mooney viscosity with the addition of amides in varying ratio which is due to the peptisation effect of amides. There was a continuous increment in the scorch time which indicates better processing safety to the reclaimed EPDM rubber waste. Results of Oscillating disc rheometer (ODR) studies are also summarized in Tables 2 - 4. As the ratio of 1,4-benzene dicarboxamides increases, the optimum cure time continuously decreases and maximum rheometric torque increases. On the basis of these resultsit is believed that 1,4-benzene dicarboxamides derived from PET waste are acting ascuring agents for EPDM rubber waste.

Table 1 – Composition of the formulations used for casting of reclaimed EPDM rubber sheets.

S. No.	Reclaimed EPDM (g)	1,4-benzene dicarboxamides (g)	Naphthalene Oil (g)	Stearic acid (g)	ZnO (g)	Sulphur (g)
1	100	1.00	2	2	0.5	1.5
2	100	1.25	2	2	0.5	1.5
3	100	1.50	2	2	0.5	1.5
4	100	2.00	2	2	0.5	1.5

Table 2 – Effect on curing characteristics and mechanical properties of reclaimed EPDM rubber sheets using 1,4-benzene dicarboxamide.

Sheet Designation	Mooney scorch time (min)	Optimum Cure time (t_{c90}) (min)	Min. TORQUE ML (dN.m)	Max. TORQUE MH (dN.m)	Mooney Viscosity	Hardness
TP1	4.2	2.0	0.45	11.61	42.0	46
TP2	4.7	1.8	0.50	12.33	41.5	47
TP3	5.0	1.7	0.55	13.04	40.3	52
TP4	5.4	1.5	0.57	13.98	40.0	58

Table 3 – Effect on curing characteristics and mechanical properties of reclaimed EPDM rubber sheets using1,4-benzene dicarbohydrazide.

Sheet Designation	Mooney scorch time (min)	Optimum Cure time (t_{c90}) (min)	Min. TORQUE ML (dN.m)	Max. TORQUE MH dN.m)	Mooney Viscosity	Hardness
TDH1	3.5	2.1	0.40	12.26	41.0	53
TDH2	3.8	2.0	0.37	12.45	40.5	55
TDH3	4.1	1.8	0.31	12.90	39.5	56
TDH4	4.6	1.7	0.27	13.45	39.0	58

Table 4 – Effect on curing characteristics and mechanical properties of reclaimed EPDM rubber sheets using*N*, *N*′-diaminoethyl 1,4-benzene dicarboxamide.

Sheet Designation	Mooney scorch time (min)	Optimum Cure time (t_{90}) (min)	Min. TORQUE ML (dN.m)	Max. TORQUE MH (dN.m)	Mooney Viscosity	Hardness
BAETP1	3.2	1.8	0.39	11.60	33.3	57
BAETP2	3.4	1.6	0.32	12.20	32.6	58
BAETP3	4.1	1.6	0.28	13.90	31.3	60
BAETP4	4.4	1.4	0.21	14.55	30.5	61

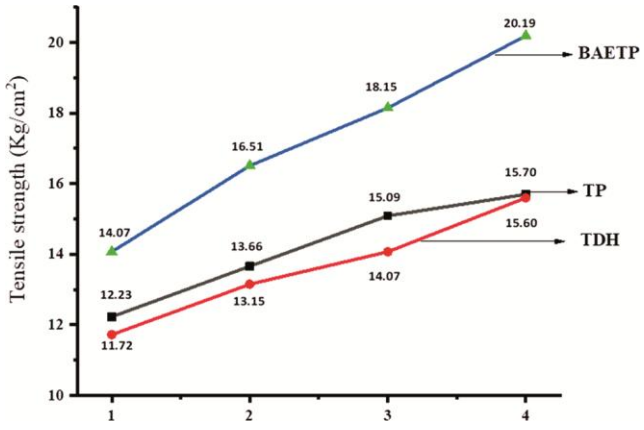

Fig. 1 – Comparative tensile strength values of casted reclaimed EPDM rubber sheets using TP, TDH and BAETP.

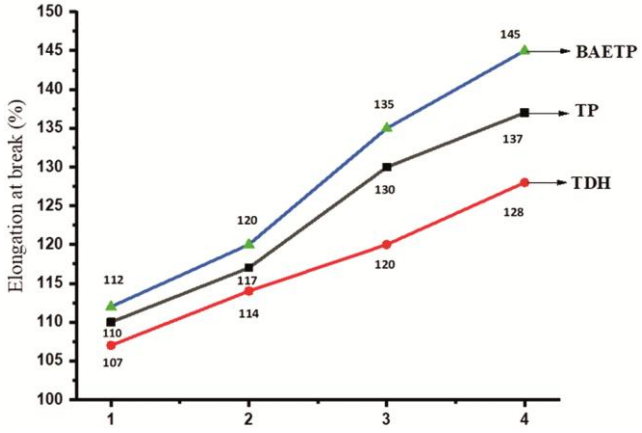

Fig. 2 – Comparative percentage elongation values of casted reclaimed EPDM rubber sheets using TP, TDH and BAETP.

3.2 Mechanical properties

Hardness values of the casted reclaimed EPDM sheets are presented in Tables 2 - 4. The comparative values of tensile strength and percentage elongation are plotted in Figs. 1 and 2.The mean values of three samples for sheet of each formulation are reported. The comparative mechanical properties presented in Figs. 1 and 2 and Table 4 shows increasing trends in tensile strength, percentage elongation and hardness which were found in the range of 12.23-20.19 kg/cm^2, 107-145 % and 46 - 61, respectively. The data reveals that, the addition of 1,4 benzene dicarboxamides in increasing ratio has improved the mechanical properties for reclaimed EPDM rubber sheets.

4 Conclusions

EPDM rubber is widely used in automotive applications such as seals, (radiator) hoses, and profiles. It is also used in roof-sheeting and seals, building and construction as profiles, as well as for electrical uses like jacketing and cable insulation. In the present study EPDM rubber waste powder is recycled successfully by using 1,4 benzene dicarboxamides derived from PET waste through chemical recycling. An attempt has been made to prepare sheets at comparatively low temperature using two roll mill and compression moulding machine. The sheets were analyzed for various mechanical properties. The tensile strength of different sheets was found in the range 12.23-20.19 kg/cm^2 and hardness values varies from 46 to 61. Percentage elongation values were enhanced significantly with increasing 1,4-benzene dicarboxamides concentration. Hence it can be stated that 1,4 benzene dicarboxamides are effective as peptisers as well as curing agents.

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