

Efficiencies of Dipolymer Rubber Blends (EPDM\FKM) using Common Weight Data Envelopment Analysis

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Polymer blends are generally categorized into two main classes: miscible blends that exist in a single homogeneous phase exhibiting synergistic properties and immiscible blends that have 2 or more different phases. There is also a third category of blends called technologically compatible blend, which exist in two or more different phases on micro scale, yet displays combination of properties. Ethylene-propylene-diene rubber (EPDM) and Hexa fluoropropylene-vinylidene fluoride dipolymer, Fluoroelastomer (FKM) blends with and without compatibilizer (MA-g-EPDM) were prepared by two-roll mill mixing. The aim of the work is to find out the best blend ratio and the amount of compatibilizer loading on thermal and mechanical properties by applying a novel mathematical programming technique called Data Envelopment Analysis (DEA). Using the different concentration of the ingredients used as inputs and the extent to which certain properties satisfied by the blends as outputs, a DEA model is developed. The blends which will be referred to as Decision Making Units (DMUs) were classified in terms of their efficiency. It is observed that the efficiency of all the compatibilized blends is higher than that of uncompatibilized blends. The maximum efficiency is obtained for 2.5 phr compatibilized blend.

Keywords: Rubber Blends, Efficiency, Data envelopment analysis.

1. Introduction

Fluoroelastomers are widely used in many industrial applications due to their excellent resistance to heat, oil and solvent^{1,2}. The increasing use of such polymers in automobiles, aerospace, off shore and energy related industries impose them stringent product performance standards under critical temperature conditions and in hostile chemical environments^{3,4}. It is also used in elastomeric sealing applications of nuclear reactors⁵. The application of fluoroether rubber was overviewed in military affairs, automobile, petroleum exploitation and semiconductor industry⁶. EPDM can also accept large amounts of filler and extender oil with no significant prejudice to the final properties. Ethylene propylene diene monomer rubber (EPDM) has excellent performance in low-temperature flexibility, thermal stability, weather ability and resistance to oxidation and ozone. Different compatibilized and non-compatibilized polar non-polar blends were prepared by different scientists⁷⁻¹⁰. Blending of FKM into EPDM can be a potential measure to prepare materials with better overall properties. However, the high incompatibility and non-co vulcanization between FKM and EPDM make it difficult to obtain a blend with better overall properties. The problem

of the cure rate incompatibility in dissimilar rubbers was studied by Rao Qiuhua et al.¹¹. Fluoroelastomer (FKM)/ ethylenepropylene-diene rubber (EPDM) blends were prepared by static vulcanization and dynamic vulcanization by Qian Lili et al.¹². The fluororubber/methyl vinyl silicone rubber (FKM/MVQ) blends elastomer was prepared by mechanical blending¹³. The mechanical properties and dynamic mechanical properties of fluoroelastomer (FKM)/ thermoplastic polyurethane (TPU) blend compatibilized with FKM-graft-maleic anhydride(FKM-g-MAH) were experimentally investigated by Dong Lijie et al.¹⁴. The thermal stability of blends depends strongly on the compatibility of the polymer. Different polymers decompose over different temperature ranges yielding different proportions of volatiles and residues. One of the most accepted methods, for studying the thermal properties of polymeric materials is the thermogravimetry¹⁵.

Maleic anhydride (MA) modification of different kinds of rubbers is a useful way of compatibilizing immiscible polymer blends as well as improving interfacial adhesion in polymeric composites. Several factors can influence mechanical properties, such as the particle size and particle size distribution of the dispersed phase, and the degree of

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adhesion between the two phases. The adequate chemical structure of the compatibilizing agent can reduce the interfacial energy between the phases and finer dispersion can be achieved¹⁵.

In the present work, the thermal stability of compatibilized and non-compatibilized EPDM/FKM blends at different blends ratios was evaluated. The effect of blend ratio and use of compatibilizer on aging resistance is also studied. The effect of ingredients in different blends is measured in terms of scorch time, hardness, heat buildup, etc is taken as outputs and the developed DEA model is applied to evaluate their efficiencies.

1.1 Basics of data envelopment analysis (DEA)

Data Envelopment Analysis is a method used to assess the relative efficiency of homogeneous group of decision making units (DMUs). This is done by measuring the efficiency based on the idea of Farrell¹⁶, which is concerned with non-parametric frontier analysis. An “efficient frontier” or a sort of “envelope” formed by a set of decision making units (DMUs) that exhibit best practices is first established in this approach and later the efficiency level to other non-frontier units is assigned according to their distances to the efficient frontier. A wide range of variations in measuring efficiency has been generated by the basic idea.

DEA models, which have wide applications in finance, health, education, manufacturing, transportation etc., are based on Linear Programming. Data Envelopment Analysis (DEA) identifies a “frontier” that is used to evaluate observations representing the performances of all the entities that are to be evaluated, by “enveloping” observations. Hence, the introduction of the term “Decision Making Unit” (DMU) was to cover, in a flexible manner, any such entity, with each such entity to be evaluated as part of a collection that utilizes similar inputs to produce similar outputs. The “degree of efficiency” thus obtained ranges between zero and unity. The DMUs (located on the “efficiency frontier”) that entered actively in arriving at these results are also identified by DEA. These evaluating entities can serve as benchmarks as they are all efficient DMUs¹⁷.

The efficiency score of a unit, which is measured on a bounded ratio scale, is the ratio of a weighted sum of its outputs to a weighted sum of its inputs. In order to maximize its relative efficiency, the weights for inputs and outputs are estimated to the best advantage for each unit. The mathematical model that underlies this is a linear program, which is given in either the multiplier form or in its dual form, the envelopment form. Whereas the former makes explicit use of the efficiency ratio, the latter gives an explicit representation of the envelope formed by the efficient frontier as well as the orientation with which the assessments are made (i.e. input or output oriented model). An output multiplied by the corresponding weight is called virtual output in terms of the multiplier form. Total virtual

output is the sum of the virtual outputs over all the output dimensions, which forms the numerator of the efficiency ratio. The definitions for inputs are analogous. The ratio of the total virtual output to the total virtual input gives the efficiency of a unit.

Let n be the number of decision making units (DMUs) of similar inputs and outputs. Let there be m inputs and s outputs. In the Classical DEA (Charnes Cooper Rhodes or CCR) model¹⁸ for evaluating the efficiency of a DMU, denoted by DMU₀ is as follows:

$$\text{Max} \sum_{r=1}^s u_r y_{rj_0}$$

Subject to constraints

$$\begin{aligned} \sum_{i=1}^m v_i x_{ij_0} &= 1 \\ \sum_{r=1}^s u_r y_{rj} - \sum_{i=1}^m v_i x_{ij} &\leq 0, j = 1, 2, \dots, n \end{aligned} \quad (I)$$

$u_r, v_i \geq 0$ for all r and i .

Where j is the DMU index, $j=1,2,\dots,n$, r is the output index, $r=1,2,\dots,s$, i is the input index, $i=1,2,\dots,m$, y_{rj} the value of the r^{th} output for the j^{th} DMU, x_{ij} the value of the i^{th} input for the j^{th} DMU, u_r the weight given to the r^{th} output, v_i the weight given to the i^{th} input, and $\sum_{r=1}^s u_r y_{rj_0}$ is the relative efficiency of DMU₀, under evaluation.

So as to compute the efficiency scores of each DMU, most of the DEA models must select a DMU, say DMU₀, among all DMUs. However, choosing different DMU₀ gives various evaluation results. Each DMU generates its hyperplane for efficiency evaluation in conventional DEA models. By the common weights approach, only one hyperplane is generated for efficiency evaluation¹⁹. Different models for deriving common weights are available now and new models continue to be explored as they are interesting from both theoretical and practical viewpoints²⁰. (A common set of weights means that only one frontier hyperplane generates a compromised solution; all DMUs lie beneath the hyperplane and agree with the final status.) Common weights that are derived by multiobjective linear programming (MOLP)^{21,22} for a DEA model are theoretically supported by the concept of Pareto efficiency. Both DEA and MOLP search for set non-inferior points. Hence, characterizing the DEA model by multi objective programming comes out to be natural reasonable and appropriate.

1.2 Common weight model in DEA

The virtual positive ideal DMU is a DMU with minimum inputs of all of DMUs as its input and maximum outputs of all of DMUs as its output^{23,24}. An ideal level is one straight line that passes through the origin and positive ideal DMU with slope 1.0, i.e. for any DMU _{j} , Δ_j^I and Δ_j^O are the horizontal and vertical virtual gaps respectively.

If we let $\Delta_j^i + \Delta_j^o$ be Δ_j and M be the maximum value of Δ_j , then using the minimum weights obtained for efficient DMUs, a new multi-objective model is given by²⁵:

Minimize

$$\sum_{j=1}^n \Delta_j + M$$

Subject to the constraints

$$\sum_{r=1}^s u_r y_{rj} - \sum_{i=1}^m v_i x_{ij} + \Delta_j = 0 \quad (\text{II})$$

$$\Delta_j \geq 0$$

$$M - \Delta_j \geq 0, j = 1, 2, \dots, n$$

$$u_r \geq \varepsilon > 0, r = 1, 2, \dots, s$$

$$v_i \geq \varepsilon > 0, i = 1, 2, \dots, m$$

Where ε is the minimum weight restriction obtained by solving the following model^{26,27}.

The normalization of inputs and outputs can be performed by using the following equations:

$$\hat{x}_{ij} = \frac{x_{ij}}{\sum_{k=1}^n x_{ik}}, i = 1, 2, \dots, m; j = 1, 2, \dots, n$$

$$\hat{y}_{rj} = \frac{y_{rj}}{\sum_{k=1}^n y_{rk}}, r = 1, 2, \dots, s; j = 1, 2, \dots, n$$

DEA efficient units will not be affected by this normalization process because CCR efficiency has a good property of unit-invariance and is independent of scale transformations of inputs and outputs. The transformed inputs meet the conditions of $\sum_{j=1}^n \hat{x}_{ij} = 1$ for $i=1,2,\dots,m$, as shown below:

Maximize ε

Subject to

$$\sum_{i=1}^m v_i = 1$$

$$\sum_{r=1}^s u_r \hat{y}_{rj_0} - \sum_{i=1}^m v_i \hat{x}_{ij_0} = 0 \quad (\text{III})$$

$$\sum_{r=1}^s u_r \hat{y}_{rj} - \sum_{i=1}^m v_i \hat{x}_{ij} \leq 0, j = 1, 2, \dots, n$$

$$u_r \geq \varepsilon, r = 1, 2, \dots, s$$

$$v_i \geq \varepsilon, i = 1, 2, \dots, m$$

where \hat{x}_{ij} ($i=1,2,\dots,m$ and $j=1,2,\dots,n$) and \hat{y}_{ij} ($r=1,2,\dots,s$ and $j=1,2,\dots,n$) are normalized input and output data. For convenience, we refer to the above LP model (III) as maximin weight model for DEA efficient units.

In the traditional DEA, ε is a given very small constant which is usually referred to as a non-Archimedean infinitesimal. However, ε in the above LP model (III) is a decision variable rather than a constant and is not necessarily very small. By solving LP model (III) for each DEA efficient unit, respectively, we can obtain a set of maximin weights, $\varepsilon_i^*, \varepsilon_{i_2}^*, \dots, \varepsilon_{i_k}^*$, for all DEA efficient units, where i_1, i_2, \dots, i_k are the labels of k DEA efficient units.

Using different amounts of ingredients, 5 types of compatibilized/non-compatibilized blends are produced. Their performance is compared based on the extent to which certain desirable properties/characteristics are satisfied. This kind of a comparison is achieved by means of a mathematical programming technique called DEA. DEA compares their performance using models II and III by calculating efficiency which is the ratio of weighted outputs (properties) to the weighted inputs.

In this paper, we find the efficiency of compatibilized and non-compatibilized EPDM/FKM blends at different blend ratio. The effect of blend ratio and use of compatibilizer on swelling, mechanical and thermal properties were studied in terms efficiency using common weight DEA.

2. Experimental Details and Analysis Using DEA

2.1 Materials

An oil-extended EPDM rubber (Keltan 7341 A), (a new CLCB grade rubber) ethylene-norbornene 7.5wt%, oil 20 phr, Mooney viscosity 53 @ 150°C, was obtained from DSM, Netherlands. Viton A401C, a fluoroelastomer containing Bisphenol curatives with specific gravity 1.82 g/cm³ and Mooney viscosity 42 @ 120°C was obtained from DuPont Dow Elastomers. Maleic anhydride grafted EPDM, MA-g-EPDM (DE5005) was obtained from DSM Elastomers, Netherlands. Zinc oxide (activator) and stearic acid (co-activator) were supplied by M/s Meta Zinc Ltd, Mumbai, and by Godrej Soaps Pvt. Ltd, Mumbai, respectively. N-Cyclohexyl benzothiazole sulphenamide (CBS) (accelerator) and tetramethylthiuram disulphide (TMTD) (accelerator) used in the present study were obtained from Polyolefins Industries, Mumbai. Sulphur (Crosslinking agent) was supplied by Standard Chemicals Co. Pvt. Ltd; Chennai. Dioctylphthalate (DOP) used was commercial grade, supplied by Rubo-Synth impex Pvt. Ltd. Paraffinic oil (processing oil) used were of commercial grade. Magnesium oxide used was commercial grade calcined light magnesia with a specific gravity of 3.6, supplied by Central Drug House Pvt. Ltd., Mumbai. High-abrasion furnace (HAF)

black (N330) used in the present study was supplied by M/s Philips Carbon Black India Ltd, Cochin. MT black (N990) was supplied by Vajra rubber products, Thrissur.

2.2 Preparation of rubber mixes and vulcanisates

In the compatibilized blends MA-g-EPDM was mixed with EPDM on a two-roll mill (16 x 33 cm²) at a friction ratio of 1:1.25. A nip gap of 0.2 mm was set at room temperature so as to get MA-g-EPDM coated EPDM. Firstly, fluorocarbon mixes were prepared by using a Brabender plasticorder at room temperature. The rotor speed and time of mixing were 60 revolutions/min. and 8 min. (This is an optimized condition for effective mixing) respectively. The compounding ingredients were added as per formulation given in Table 1. MA-g-EPDM coated EPDM and FKM (previously mixed) blends were prepared on laboratory size two-roll mixing mill and the temperature maintained at 70 ± 5 °C. The duration of the mixing time is 20 minutes. The compounding was done as per in ASTM D 3184-89, 2001. Detailed experimental technique is given in our published research article²⁸.

2.3 DEA efficiencies of non-compatibilized and compatibilized rubber blends

The blends were designated as follows. E₁₀₀ means EPDM and E₀ means FKM. E₉₀ means a blend of 90 phr of EPDM and 10 phr of FKM. The binary blends were designated as E₁₀₀, E₉₀, E₈₀, E₇₀, E₆₀, E₅₀ and E₀. The Table 1 and Table 2 shows the Input and output values against blend ratio respectively. The compounding ingredients used in the blend are considered as inputs. The effect of different blend on properties like Scorch time, Heat buildup, CD in toluence

etc... are considered as outputs. Based on these inputs and outputs performance of the blends are analyzed where the different blends are taken as entities (Decision Making Units - DMUs) which are to be compared.

The corresponding efficiency values using the developed common weight data envelopment analysis model are given in Table 3 and in Fig. 1. In the case of an un-compatibilized blend, the efficiency of the blend increases with increase in the addition of FKM rubber. From the table, it is very clear that E₅₀ has the maximum efficiency. This is in good agreement with the mechanical and thermal properties studied.

The Crosslink density (CD) of all samples is given in Table 2. CD of any vulcanizate has to be measured in a good solvent (good solvent is a solvent in which the polymer vulcanizate shows maximum swelling). But when the swelling experiment is done in the case of polymer blends (one polar and other non-polar) the swelling behaviour in two solvent (one a good solvent for EPDM and other a good solvent for FKM) is different. The crosslink density determined experimentally by swelling will not give the exact crosslink density of the blend. But in the case of a blend like EPDM/FKM there is no solvent which is good for both. So this experiment was done in both toluene and MEK. Toluene is a good solvent for EPDM and MEK is a good solvent for FKM. All compatibilized blends display lower solvent uptake than the non modified blend, which is an indication of increase in crosslink density.

The hardness of all samples is given in Table 2. Hardness increases with increase in FKM content. The highest hardness is obtained for E₀, 70 Shore A. The lowest hardness is obtained for E₁₀₀, 60 Shore A. In the case of blends the hardness is in between the pure compound. The stiffness of the FKM

Table 1. Compounding ingredients as inputs for Non-compatibilized Blends in DEA analysis

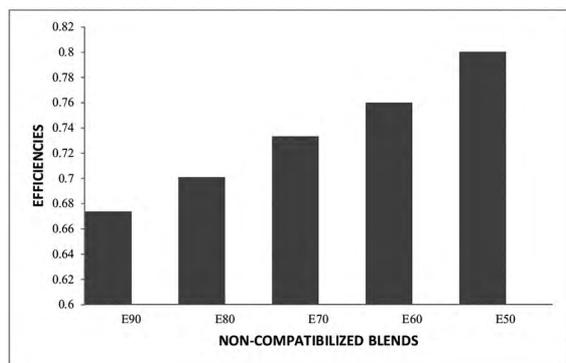
	INPUTS									
	EPDM (phr)	FKM (phr)	ZnO (phr)	Stearic Acid (phr)	HAF (phr)	Paraffinn oil (phr)	DOP (phr)	CBS (phr)	TMTD (phr)	S (phr)
E90	90	10	4.05	1.35	18	5	5	0.9	0.9	1.35
E80	80	20	3.6	1.2	16	5	5	0.8	0.8	1.2
E70	70	30	3.15	1.05	14	5	5	0.7	0.7	1.05
E60	60	40	2.7	0.9	12	5	5	0.6	0.6	0.9
E50	50	50	2.25	0.75	10	5	5	0.5	0.5	0.75

Table 2. Effect of blend ratio on properties as outputs for Non-compatibilized Blends in DEA analysis

	OUTPUTS									
	SCORCH TIME (min)	HARDNESS (Shore A)	HEAT BUILDUP (°C)	C D IN TOLUENCE (g mol/cm ³)	C D IN MEK (g mol/cm ³)	T O C (°C)	T 50 C (°C)	T 5% (°C)	T 15% (°C)	E (KJ/Mol)
E90	2.68	53	14	10.2	17.8	410	480	258	357	112.86
E80	2.6	55	16	11.4	14.1	420	481	259	358	118.53
E70	3.23	56	17	11.6	10.3	423	481	260	361	119.46
E60	2.77	62	19	12.6	7.4	421	482	262	363	120
E50	2.37	65	21	13.6	5.8	422	490	263	381	121.22

Table 3. The efficiencies of different Non-compatibilized Blends using DEA model

Blends	E ₉₀	E ₈₀	E ₇₀	E ₆₀	E ₅₀
Efficiency	0.67391	0.70103	0.73335	0.76017	0.80065

**Figure 1.** Efficiencies of different Non-compatibilized Blends

is higher compared to EPDM. There is a gradual increase in heat generation values of all the blends (Table 2 and 4). The energy dissipation can be through loss at filler-matrix interface, friction between the chains and break down of filler structure. Compared to uncompatibilized blends, the compatibilized blends show higher heat build-up values. This will be manifested as lower resilience values. The highest heat build-up is obtained for E₀, 8°C. The lowest hardness is obtained for E₁₀₀, 19°C. In the case of blends the heat build-up is increases when compared to the pure compounds. This is due to the increased stiffness of the inter-molecular chains between the two dissimilar blends.

Table 4. Compounding ingredients as inputs for Compatibilized Blends in DEA analysis

	INPUTS										
	EPDM (phr)	FKM (phr)	MAg-EPDM (phr)	ZnO (phr)	Stearic Acid (phr)	HAF (phr)	Paraffinn oil (phr)	DOP (phr)	CBS (phr)	TMTD (phr)	S (phr)
E501	49	50	1	2.25	0.75	10	5	5	0.5	0.5	0.75
E502.5	47.5	50	2.5	2.25	0.75	10	5	5	0.5	0.5	0.75
E505	45	50	5	2.25	0.75	10	5	5	0.5	0.5	0.75
E505*	50	50	5	2.25	0.75	10	5	5	0.5	0.5	0.75
E5010	40	50	10	2.25	0.75	10	5	5	0.5	0.5	0.75

Table 5. Effect of blend ratio on properties as outputs for Compatibilized Blends in DEA analysis

	OUTPUTS										
	SCORCH TIME (min)	HARDNESS (Shore A)	HEAT BUILDUP (°C)	C D IN TOLUENCE (g mol/cm ³)	C D IN MEK (g mol/cm ³)	T 0 C (°C)	T 50C (°C)	T 5% (°C)	T 10% (°C)	T 15% (°C)	E (KJ/Mol)
E501	2	66	26	14.3	6.2	433	498	261	327	416	125.98
E502.5	2.13	65	29	16.4	6.5	440	495	262	326	411	128.54
E505	2.14	65	29	18.7	7.3	436	502	271	342	427	131.02
E505*	2.32	66	28	19	7.9	435	498	265	327	421	133.98
E5010	2.4	66	30	18.8	8.4	427	496	260	332	424	126.43

The MA-g-EPDM compatibilized E₅₀ blends were designated as E_{50X}, where X=1, 2.5, 5, 5* and 10. X denotes the weight percentage of the compatibilizer in the blend. The input and output values given in Table 4 and Table 5 are used to find the efficiencies of E₅₀₁, E_{502.5}, E₅₀₅, E_{505*}, E₅₀₁₀. “*” indicates that MA-g-EPDM as an additive (compatibilizer) at 5 phr (parts per hundred rubber).

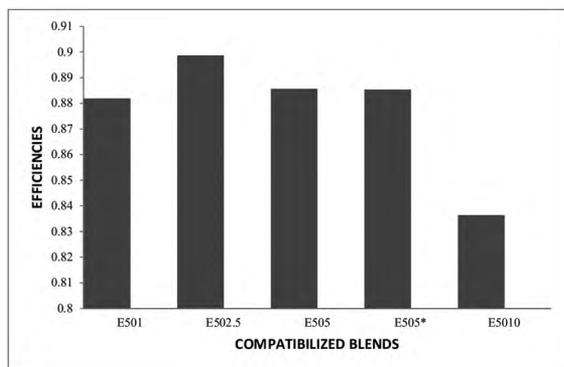
These efficiency values using the proposed model are given in the Table 6 and in Fig. 2. It is clear that the compatibilized blends show higher efficiencies compared to uncompatibilized blends. The increment of efficiency of compatibilized blend compared to uncompatibilized EPDM/FKM blends is due to the formation of hydrogen bonding and improvement in the interfacial interactions between EPDM and FKM in the presence of compatibilizer, as confirmed by the mechanical properties.

2.4 Discussion and analysis

The scorch time is slightly increased by the addition of FKM to EPDM rubber. But the addition of compatibilizer, there is no significant variation in scorch time (Table 2 and Table 5). The scorch time (T₁₀) is expressed in minutes. In all the blends the scorch time is approximately 3 minutes. The Table 5 shows the hardness and heat buildup of the vulcanizates. The hardness increases with increase in FKM content. In all the cases the compatibilized vulcanizates show better properties confirming the effect of compatibilization. There is a gradual increase in heat generation values of all the blends. The energy dissipation can be through loss at filler-matrix interface, friction between the chains and break down of filler structure. This will be manifested as

Table 6. The efficiencies of different Compatibilized Blends using DEA model

Blends	E_{501}	$E_{502.5}$	E_{505}	E_{505^*}	E_{5010}
Efficiency	0.88194	0.89874	0.88194	0.88537	0.83643

**Figure 2.** Efficiencies of different Compatibilized Blend

lower resilience values. As given in Table 2, the resilience values show a linear decrease with increase in FKM content. Compared to uncompatibilized blends (refer Table 2), the compatibilized blends (refer Table 4) show higher heat build-up values. The variation in mechanical properties is in good agreement with the efficiencies by DEA analysis.

The swelling percentage is the measurement of the degree of cross linking, the reduction in swelling indicating increase in cross link density and thus the reduction in solvent uptake. The increase in cross link density of compatibilized blends may be due to the hydrogen bonding with MA-g-EPDM and FKM rubber. The extent of swelling of a blend in a solvent depends on the structure of the polymer phases and can be related to the properties of the polymer chains, such as molecular mobility, phase interaction etc. and also related to the vulcanization procedure of rubber blend. The compatibilized blends are vulcanized for the second time (post curing) and equilibrium swelling is reduced more after aging than that of the uncompatibilized blends. All compatibilized blends display lower solvent uptake than the non modified blend, which is an indication of increase in crosslink density (Table 5). This is illustrated with the help of the developed mathematical model.

The initiation of degradation (T5%) of EPDM is found to occur at 255°C and that of FKM at 450°C²⁷. In the case of uncompatibilized blends, the incorporation of FKM shows only a slight improvement in the initiation temperature of degradation. But in the case of all compatibilized E_{50} blends, the incorporation of FKM is found to shift the degradation temperature to a higher region. The variation in thermal properties is in good agreement with the efficiencies by DEA analysis. The mentioned facts are depicted in the graphs given below:

3. Conclusions

Data Envelopment Analysis is used to estimate the relative efficiency of homogeneous group of decision making units (DMUs). *Efficiency in this context is the extent to which these DMUs possess the properties.* The developed mathematical programming based model is successfully applied in EPDM/MA-g-EPDM/FKM blends. The efficiencies of different blends were monitored and are in good agreement with the experimental results. In the case of uncompatibilized blends, the maximum efficiencies are obtained for 50:50 EPDM/FKM blends. The minimum swelling properties and maximum physical and thermal properties are obtained for this blend. The experimental results are again confirmed by DEA programming. In the second part of the work, the DEA model is applied for MA-g-EPDM compatibilized blends. The efficiency of all the compatibilized blends is higher than that of uncompatibilized blends. The maximum efficiency is obtained for 2.5 phr compatibilized blend. It is also observed that there is less variation in the efficiencies of compatibilized blends as there is not much difference in the amounts of ingredients present in these blends. But it helps us to identify the best blend as 2.5 phr compatibilized blend.

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