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Polyurethane Flexible Foam Fire Behavior

Ahmadreza Gharehbagh and Zahed Ahmadi

Additional information is available at the end of the chapter

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1. Introduction

Polyurethanes are a broad range of polymers, which are formed from the reaction between diisocyanates or polyisocyanates with diols or polyols. According to **the types and amounts of, polyols, isocyanate, ingredients** and the overall reaction circumstances, a broad range of products **like flexible foams, rigid foams**, elastomers, coatings and adhesives are produced.

Since the polyurethane products specially foams are playing an indispensable rule in our daily life **because of wide range of applications in automotive, household, refrigerators, insulations**, reducing of the fire risk of such a products are become more vital.

Conventional polyurethane **flexible** foams are easily ignited by a small flame source and burn rapidly with a high rate of heat release and smoke and toxic gases. This high flammability of polyurethane flexible foam is related to its cellular and open cell structure and low density of such foams. Oxygen can easily pass through the cells of the combustible material and in subjecting with an accident, smoldering cigarette or an electrical shock, foam catch fire [1].

Polyurethanes can be resisted against fire by different ways. **Depends on the types and applications of them, fire resisting could be done by the flame retardants using or by changing in the polymer structure.** In the whole picture the polymer ignition can be controlled by the following **factors**.

1. Extinguishing material reduction
2. Air supplying source reduction
3. Fire diffusion and heat generation reduction
4. Increasing of the energy needed for entire combustion process

Different types of the fire retardants could be used according to one of the above mentioned categories. The flame retardants are acting according to one of the following mechanisms.

1. Reaction with the flame and preventing of the spreading of the fire by the created free radical blocks.
2. Preventing of the oxygen diffusion into the polymer
3. Lowering of the flame temperature with **removing energy from the system**
4. Char creation and creating a free place between the solid polymer and the disposed area.
5. Polymer expansion and making a free place between the fire and the decomposed polymer.

There are lots of materials which are known as the fire retardants according to the following groups.

- Halogenated flame retardants which are acting in the gas phase with disturbing the hydrogen-oxygen reaction. They react with hydrogen and create the halogen free radicals then they block free radicals of decomposed polymer.
- Metal oxides which act in solid or gas phase and some of the members of this group cause to reduce the flame temperature.
- Phosphorous containing compounds which create char on the extinguishing area of the polymer and prevent the oxygen feeding to the flame.
- Halogen free FR which the two main candidates are EG and Melamine. The heat stability of the polyurethanes especially the rigid foams at high temperatures depend on the isocyanurate to allophanates and biurets cross-linked bond ratio. Carbodiimide is produced by the condensation reaction of isocyanate with lose of CO₂ (Fig.1). This reaction can be catalyzed by the cyclic phosphine-oxide. Generated carbodiimide is used as an anti-hydrolyze agent in the polyurethanes. The heat stability of the diverse products of polyurethanes is classified in Table (1).

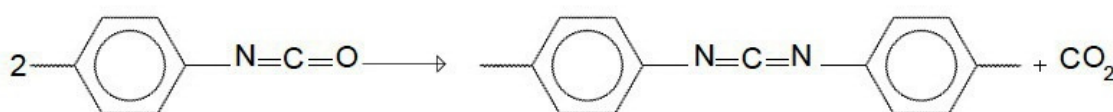


Figure 1. Condensation reaction of isocyanate to make Carbodiimide

Bonds	Decomposition Temperature(°C)
allophanate	100-200
biuret	115-125
Urea	160-200
Urethane	180-200
Substituted Urea	235-250
carbodiimide	250-280
Isocyanurate	270-300

Table 1. Heat stability of the diverse products of polyurethanes

1.1. Polyurethane foam morphology

Polyurethane morphology plays a vital rule on the fire properties of the polyurethane foam. The porous structure of the foam helps to diffuse the oxygen easily in to the foam and accelerate the ignition process. Fig.2 shows the SEM Picture of the polyurethane flexible foam with no filler inside. As it is clear, the cell structure of the foam includes Cell window, Strut and Strut joint [1].

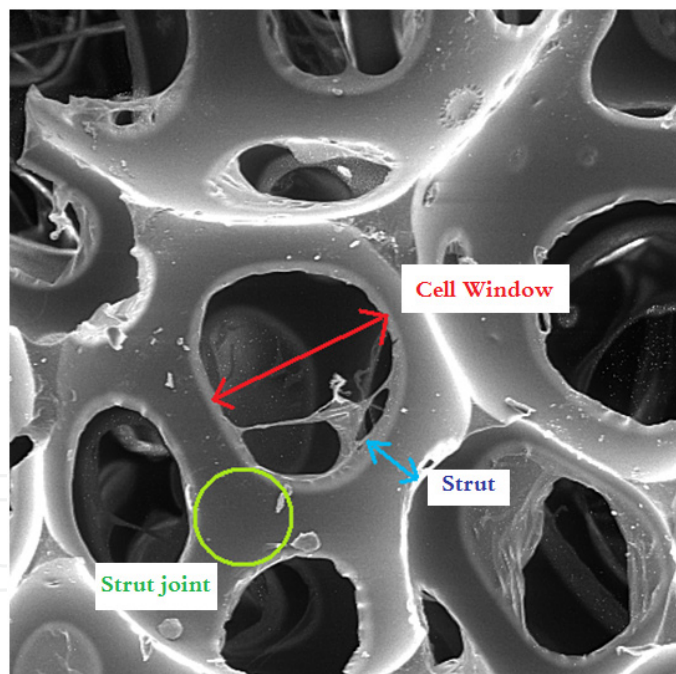


Figure 2. Fundamental concepts of polyurethane foam Cell Structure (SEM×200)

1.2. Polyurethane flexible foam fire retardants

1.2.1. Halogenated phosphorous flame retardants

In recent years the phasing out of some types of halogenated FR (flame retardant) due to persistence at environment and bioaccumulation and toxicity has been more investigated.

TMCP (Tris (2-chloroisopropyl) phosphate) and TDCP (Tris (1, 3-dichloroisopropyl phosphate) are two well-known liquid FR which are used in polyurethane flexible foam to make fire resisted (Figure3-4). Table (2) illustrate some important parameters of the mentioned fire retardants. [2]

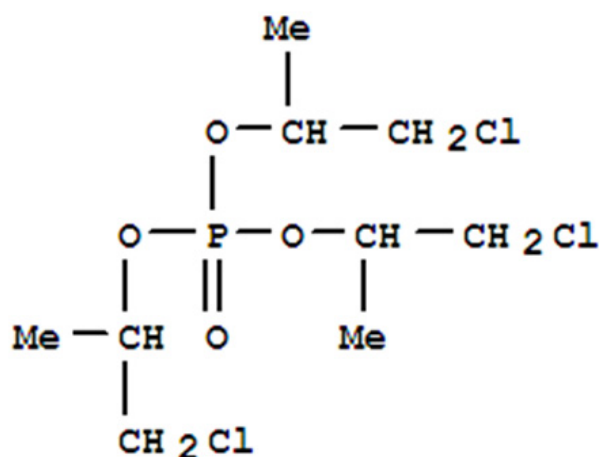


Figure 3. Chemical structure of TMCP

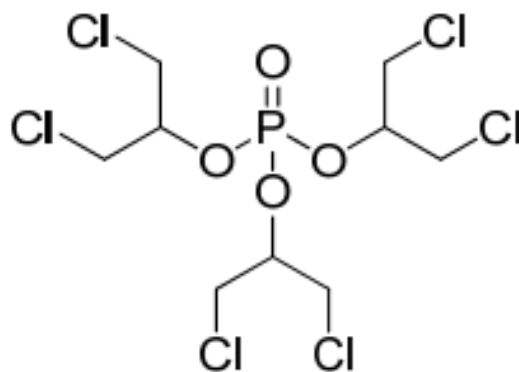


Figure 4. Chemical structure of TDCP

Property	TMCP	TDCP
CL content (%)	32.5	49
Mw(g/mol)	327.55	430.91
P content (%)	9.5	7.1
physical state at 25°C	clear liquid	clear liquid
Water solubility(%)	< 0.05	< 0.05

Table 2. Properties of TMCP and TDCP

Studies show that in the foams with only liquid FR (TMCP , TDCP) a very divergent combustion behaviour has been indicated. TMCP containing foams show lower TWL(total weight loss) and shorter burn time compared to TDCP containing foams. Moreover, TMCP containing foams didn't show any significant dripping and subsequent hole formation, a phenomenon seen at all levels of TDCP addition. TMCP and TDCP addition leads to decrease in the THE(total heat evolved) but an increase in the amount of smoke and carbon monoxide produced and this is why normally some amount of other FR such as melamine is added to the TMCP and TDCP containing foams to decrease total heat evolved, total smoke produced and CO emission significantly[2,3].

1.2.2. Halogen-free flame retardants

Due to the above mentioned reasons it has been a driving force to move toward the halogen free FR to compensate those weakness of halogenated FR, **despite of some disadvantages that the halogen free FR have e.g. they are mostly in solid state and they show process difficulties.** There are different types of halogen-free flame retardants which are behaving with different mechanisms. First group acts according to the expansion inside the polymer and oxygen-diffusion prevention and second group does by the cooling of the ignited surface of the polymer. One important example of the above mentioned groups are leaded by expandable graphite (EG) and Melamine powder respectively.

1.2.2.1. Expandable graphite

EG is a graphite intercalation compound in which some oxidants, such as sulfuric acid, potassium permanganate, etc. are inserted between the carbon layers of the graphite [4]. Fig.5 illustrates the chemical structure of Graphite, diamond and C60 [1].

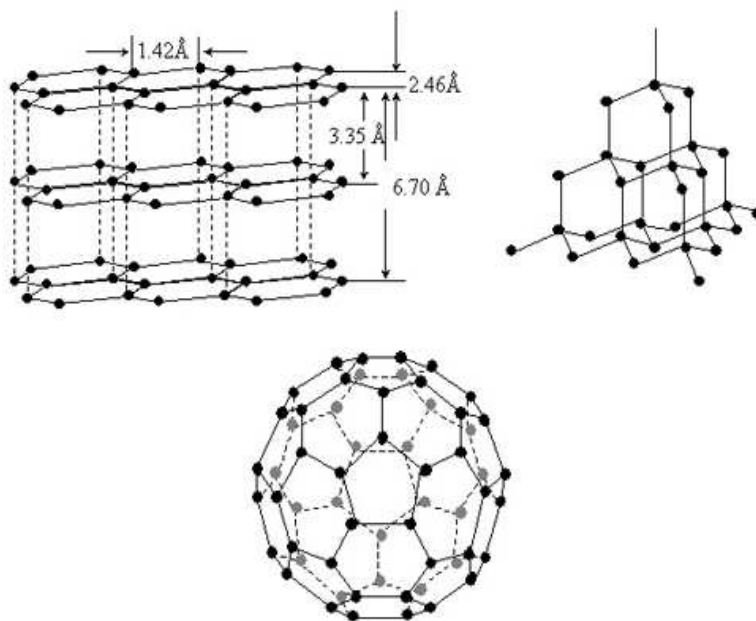
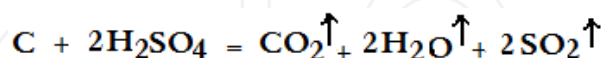


Figure 5. Comparison of Lattice graphite, Diamond and C60

When EG is subjected to a heat source, it expands to hundreds of times of its initial volume and creates voluminous, stable carbonaceous layer on the surface of the materials. This layer limits **the heat transfer from the heat source to the substrate and the mass transfer from the substrate to the heat source resulting in protection of the underlying material** [5, 6]. **The redox process** [7] between Sulfuric acid and graphite generates the blowing gases according to the reaction:



The fire retardancy of EG is done by two steps [1]:

- The EG expands under the impact of Heat up to about 500 times of its original volume and creates a very large surface. It allows a quick oxidation of the carbon. The oxygen is taken out of the air and makes the air almost inert. This inert air extinguishes the fire.
- EG doesn't create flames while oxidation and will extinguish if no more heat will be applied to the glowing graphite. Therefore, no source of fire will be generated by the oxidizing graphite.

The more characteristic factors for EG which should be considered are:

- SET (start expansion temperature)
- Expansion volume
- Strength

Figure (6, 7) show particle size and distribution of two types of EG with different sizes (0.18mm, 0.25mm)

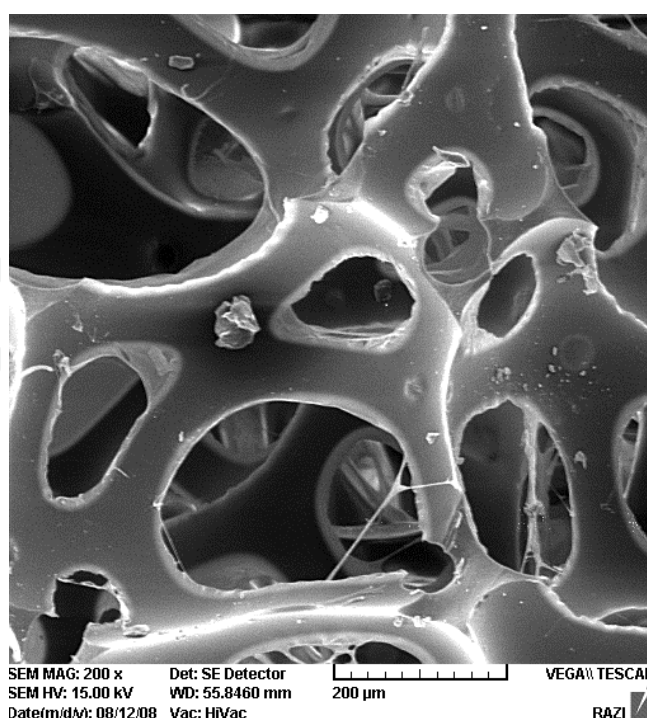


Figure 6. Particle size and distribution of 8% of EG (0.18mm) (SEM ×200)

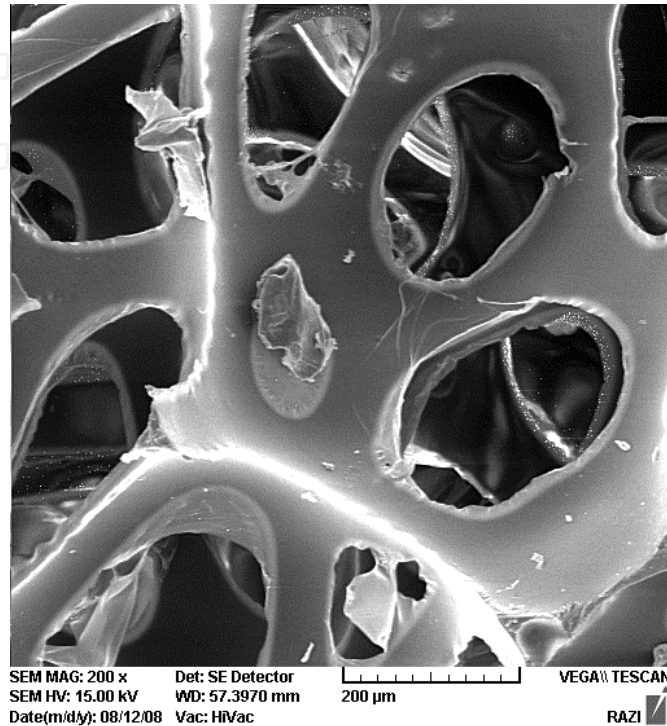


Figure 7. Particle size and distribution of 8% of EG (0.25mm) (SEM×200)

1.2.2.2. Melamine

Melamine acts as fire retardant and smoke-suppressant according to the following combined mechanisms [8].

- Melamine is believed to act as a heat sink, increasing the heat capacity of the combustion system and lowering the surface temperature of the foam. Thus the rates of combustible gas evolution and burning are reduced.
- The nitrogen content of the melamine may partly end up as nitrogen gas when melamine burns, providing both a heat sink and inert diluents in the flame. The presence of melamine in the foam results in less heat generated by the flame, consequently less heat fed back to the foam and the rate of foam pyrolysis, i.e. generating of volatile fuel is reduced.
- Due to a chemical interaction between melamine and the evolved isocyanate fraction creating from degradation of polyurethane foam. This interaction reduces the amount of diisocyanate the main contributor to the smoke and CO release (Fig.8).

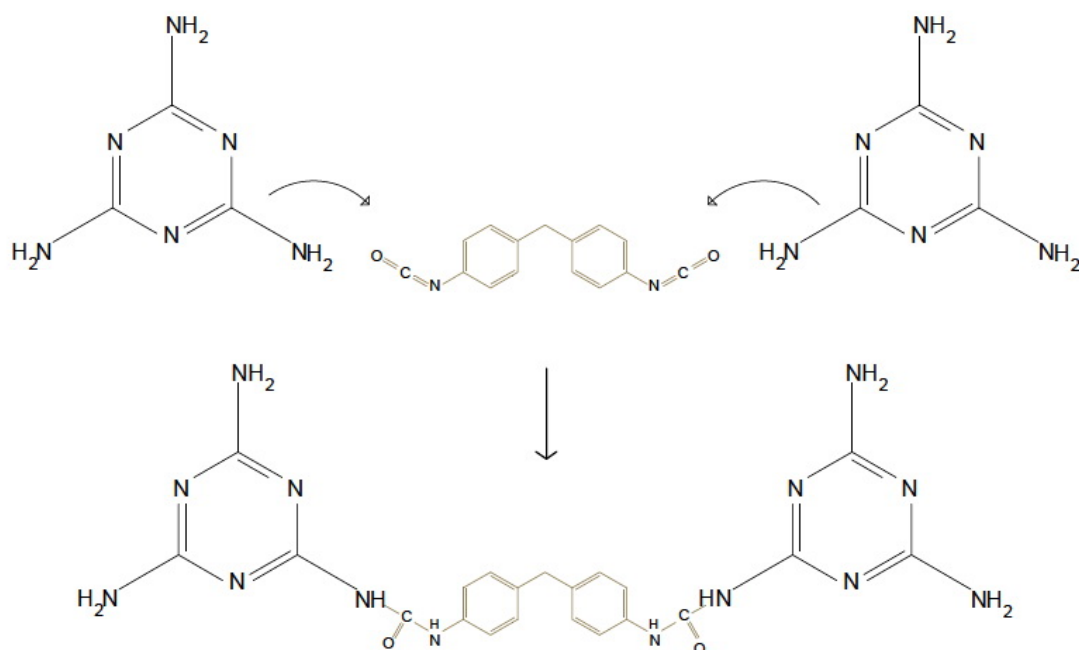


Figure 8. Chemical reaction between melamine and diisocyanate (MDI)

Figure (9) shows particle size and distribution of melamine powder inside the flexible foam.

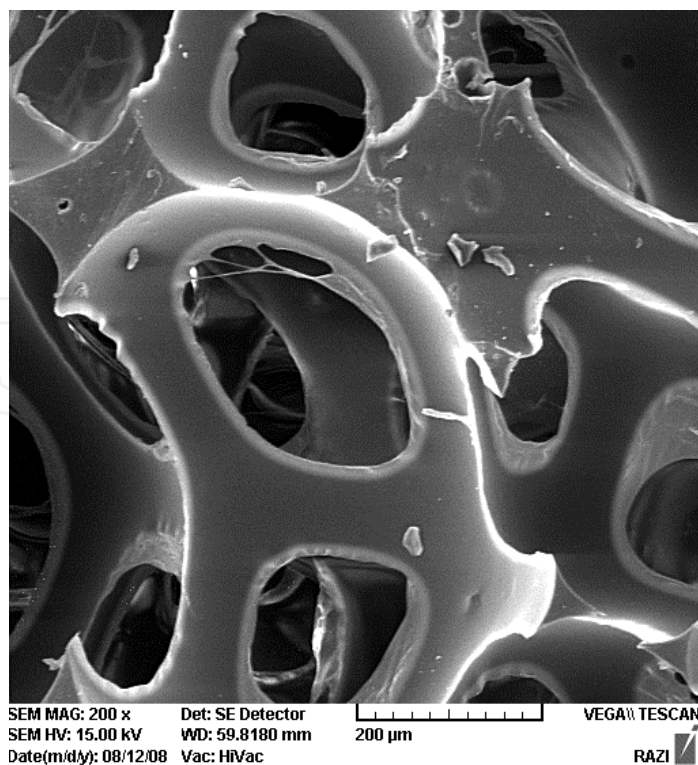


Figure 9. Particle size and distribution of 8% of melamine (SEM ×200)

1.3. Properties of the polyurethane flexible foam with different types of fire retardants

Comparison between halogenated flame retardants which are mainly liquid with halogen free flame retardants (expandable graphite and melamine powder) which both are solid can be categorized as **four** items.

- Processing
- Reactivity
- Fire properties
- Physical properties.

1.3.1. Foaming process

The best choice for the processing as it is clear will be the liquid grade which has a good dispersion inside the polyol and less side effects.

Expandable Graphite has a limit pot life (3-4 hours) and when it subjects with the polyol component, it attacks to the catalyst of the polyol and destroys the catalyst during the foaming process. **For the foam producing, the highly recommendation is the EG containing polyol should react with proper isocyanate before the EG pot life reaches or the EG should be injected by an individual stream and mix with the polyol stream in the mixing head instead of pre-mixing with polyol.** Otherwise the produced foams will be collapsed. The other disadvantage of this technology is related to the fact that EG is very corrosive and make the mixing head to be damaged and it is preferred to be used a hardened grade of mixing head, a damaged mixing head needle picture is showed in (Fig.10)[9].

The advantages of this technology is the good homogeneity of the EG particles inside the polyol.



Figure 10. Mixing head needle corrosion by EG

Despite the EG, melamine has the longer pot life inside the polyol, which is around 24 hours but the fast sedimentation of the melamine powder in the polyol will be the main disadvantages so we need a suitable method to disperse the melamine powder in the polyol very well to achieve a homogeneous mixture.

1.3.2. Reactivity

Foam reactivity is determined by the following parameters:

- Cream time (sec): Cream time is the time when the polyol and isocyanate mixture begins to change from the liquid state to a creamy and starts to expansion subsequently.
- Gel time (sec): Gel time is the time the foam start to stiffen
- Rise time (sec): rise time is the time the foam reach to its maximum height
- Recession factor (%): the height percentage the foam is settled after 5 min after the rise time
- Expansion factor (cm/kg): the proportion of the maximum height of the foam to foam weight.

The flame retardants would affect on the foam reactivity depend on the types of them, whether they are solid or liquid. Because they make changes in cell structure and total system heat capacity. The recession factor goes up with addition of EG and melamine but with different slopes. This is due to the increase in the average cell size of the foam. The bigger the flake size, the larger the cells and higher the recession factor. On the other hand melamine powders with small sizes are embedded on struts and joints and increase the viscosity and reduce the drainage rate which consequently, decreases the number of cells with bigger sizes [10]. Melamine powders with bigger size (bigger than struts and joints) are embedded inside the cell walls and open the cells.

Expansion factor which is related to the free rise density (FRD) reduces with addition of the EG and melamine in the foam. This is due to the increase the heat capacity of the entire system because of high heat capacity of melamine and EG. When melamine and EG content increases in the system, the heat capacity of the system increases and the system temperature reduces, therefore, the foam height and consequently expansion factor reduces [11].

1.3.3. Fire properties

The fire properties of the polyurethane flexible foams have been evaluated by different types of methods depends on the customer requirements. For example, the automotive, railway and airplane industries have their own standards.

The most important parameters which have been tested are: Cone calorimetry, flammability, smoke density and toxicity.

1.3.3.1. Cone calorimetry ISO 5660

The principle of the calorimetry by oxygen consumption (cone calorimeter) is based on the relation between the oxygen consumption and the heat release during the combustion. The

ratio between the heat release and the weight of oxygen consumed is a constant (Huggett constant) equal to 13100 kJ/kg. It has been previously demonstrated that cone calorimeter results are in good correlation with results obtained in full scale fire test on upholstered furniture [3].

Samples of flexible foams (10*10*5cm) were exposed in a Stanton Redcroft Cone Calorimeter according to ASTM 1356-90 under a heat flux of 35kW/m² (case of fully involved real fires). This flux was chosen because it corresponds to the evolved heat during a fire. An electrical spark igniter ignited volatile gases from the heated specimen. At least three specimens have been tested for each formulation. Data were recorded with a computer connected to the cone calorimeter. The test gives the opportunity to evaluate:

- RHR: Rate of Heat Release (kW/m²)
- Figra: fire growth rate: RHR/time (kW/m²/s)
- Weight loss (wt. %)
- Emission of carbon monoxide (ppm)
- TVSP: Total volume of smoke production (m³)
- THE: total heat evolved (kJ/cm²/g)

The combustion of flexible polyurethane foams is a two steps process (Fig.11).

The first step corresponds to the melting of the foam into a tar and the second to the combustion of the tar previously produced. [3]

These two degradation steps lead to two distinct peaks of rate of heat released.

- The RHR1 values (the values of RHR of the first and second RHR peaks).
- The T1 and T2 values (times at which RHR1 and RHR2 occur).
- The Figra2 values (the two maximum peaks on the Figra curve).

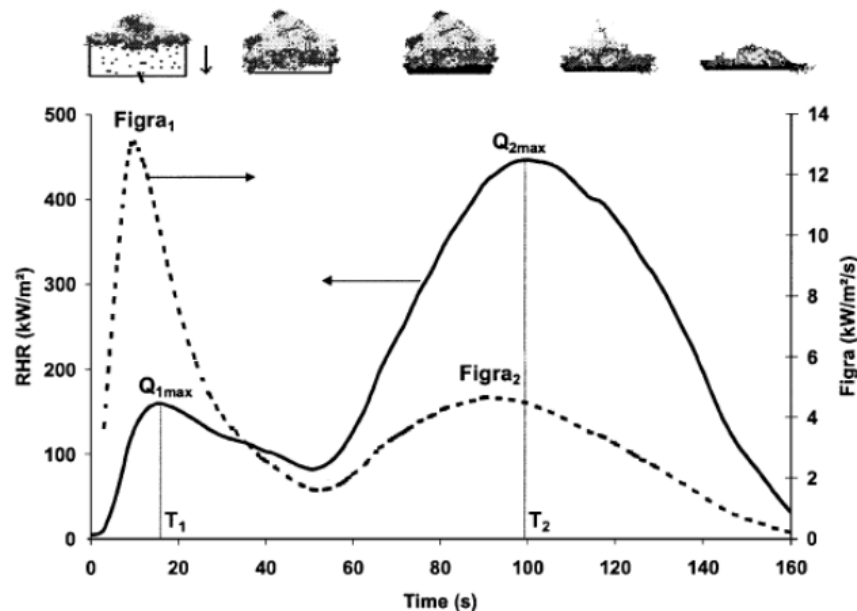


Figure 11. Combustion of flexible polyurethane foams: a two-stage process

1.3.3.2. Flammability

Flammability of the polyurethane foam is running with wide range of test methods depends on the applications and customers specification. Some fire tests standards include: FMVSS NO.302, British Standard 5852, ISO 9772 and FAA/JAA 25.853 Appendix F. as an example the airplane seat foam fire tests according to FAA/JAA 25.853 Appendix F have been investigated.

In this test 5 samples with 75mm*305mm*13mm dimension have been subjected with flame vertically for 12 sec and the following parameters have been investigated.

Burning time (the time that burning is continuing after removing the flame source) Burned length (the length of the foam which is damaged by the burning process) Time of dripping (the time which droplet continues to burn).

Synergetic effect

The synergetic effect of different types of FR has been observed. for instance, the fire properties of the EG loaded foams is much worse than when it is used by mixing with a liquid FR such halogenated phosphorous flame retardant. **Also when some amount of melamine is added to the TMCP and TDCP containing foams it helps to decrease total heat evolved, total smoke produced and CO emission significantly[2].**

Also the mixing of the liquid FR could boost the fire properties of the melamine loaded foam considerably.

1.3.3.3. Smoke density and toxicity

Smoke density and Toxicity are measured according to Airbus Directive ABD0031 (2005) on two categories:

1. Flaming
2. Non-flaming

Samples with 76mm*76mm*13mm are chosen to do the above mention tests against them in flaming and non-flaming status.

1.3.4. Physical properties

Physical and mechanical properties of the flexible polyurethane foams are evaluated by different types of tests in order to make an entire picture from the foam part performance during the consuming by the customer. For instance, flexible polyurethane foam is widely used as car seat foam and it has to keep its shape and other properties such as hardness and compression set during the time which is used. The most important properties of the polyurethane flexible foam as car seat foam according to RENAULT specifications are viewing as below.

- Core Density
- Compression Load Deflection (CLD: P25/5) and Sag-Factor according to D411003

- Compression Set according to D451046
- Tensile strength & Elongation at break according to
- Tear Strength according to D411048
- Resilience in 1st and 5th cycle according to D455128

When the polyurethane flexible foam is going to be fire resisted, some fire retardants in liquid or solid forms are entered in to the foam structure and make some changes in the physical properties of the final foam part. Mostly the valuable changes have been observed by the solid FR addition rather than the liquid one.

Depending on the fire retardant nature, shape and size, their addition may have some positive or negative effect on foam physical-mechanical properties. By loading the solid FR with the same amount, the foams become softer, because both additives have a similar size as cell windows and make the foam inhomogeneous. With EG, the homogeneity would be less than the foam loaded by melamine, because of its bigger size and flake shape which makes the foam much softer [1].

Sag-factor or the comfort index [12] increasing when the percentage of EG and melamine increases. It means that by adding solid FR, the comfort index would change considerably. Compression set, which is another very important factor, has increased by rising the EG percentage, but there was almost no changes in CS by increasing the melamine content. This effect is due to destroying effect of the cells structure by both additives but mainly by the EG.

Tear strength of the foams has improved by increasing the EG which could be related to the rigidity of EG flakes but deteriorates when melamine is added.

Finally, the resilience in 1st cycle is decreased for all additives but it is recovered in 5th cycle, because in 1st cycle the polymer chains have lost their flexibility due to rigid particles but after 5 cyclic movements the particles are embedded in struts and joints and the foam restores its flexibility.

1.4. Statistical method

Principle component analysis (PCA) is a useful method to illustrate relations between different parameters by using STAT-BOX-ITCF [13, 14].

Interpretation of the results consists first in the checking the representation of the variables in the circles of correlation. The correlations between variables are deduced from the relative position and the length of their corresponding vectors on the circle of correlation. An example of interpretation is done in (Fig.12); the angle between two vectors defines the intensity of the correlation (vectors 1 and 5). If α is $=90^\circ$, no relation exists between the variables. The strength of the correlation is higher when the angle is close to 0° or 180° . So, orthogonal vectors (vectors 1 and 2) mean no correlation between the variables. Data are strongly correlated if their vectors are collinear (vectors 1 and 3, and vectors 1 and 4). The nature of the correlation also depends on the direction of the vectors: if vectors have the

same direction (vectors 1 and 4) the variables are correlated, i.e. an increase in the variable linked to the vector 1 corresponds to an increase in the variable linked to the vector 4. Inversely, if vectors are opposite (vectors 1 and 3), the variables are anti-correlated.

The correlation between two variables is also a function of the length of the vectors. As example, vectors 2 and 6 are co-linear and so should be anti-correlated. But the weak length of the vector 6 means that its corresponding variable does not influence the variable linked to vector 2 [2].

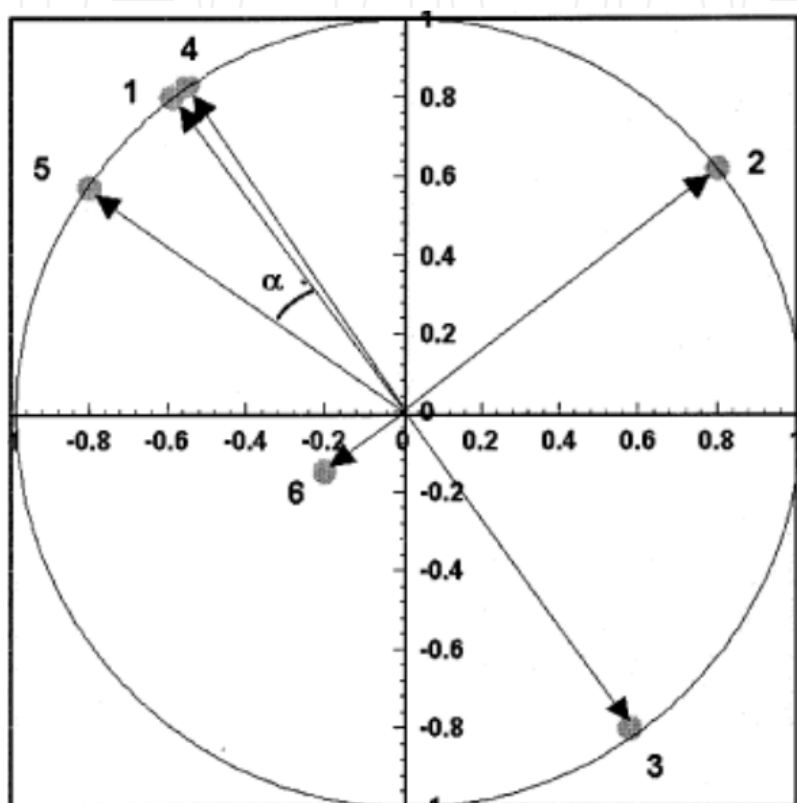


Figure 12. Interpretation of principal components analysis

1.4.1. Cone calorimeter–FMVSS 302

The principal components analysis from cone calorimeter and FMVSS 302 data shows the following correlations (Fig.13)

- *RHR1* is moderately correlated with *Figra1*: *Figra1* is a variable that depends on the first peak of HRR (also called q_{1max}), d the time it occurs. So, it seems quite coherent to find this kind of relation if the relative variation of the time is low.
- *RHR1* is correlated with *Figra2*. In the cone calorimeter, the foam degradation occurs in two main steps. It is obvious that an important consumption of fuel in the first step leads to a lower *Figra2*.
- FMVSS is strongly correlated with *Figra1* and *Figra2* and inversely correlated with *RHR2*. The lower *Figra1* and *Figra2*, the slower the flame spread. A high *RHR2* means loss of heat by dripping.

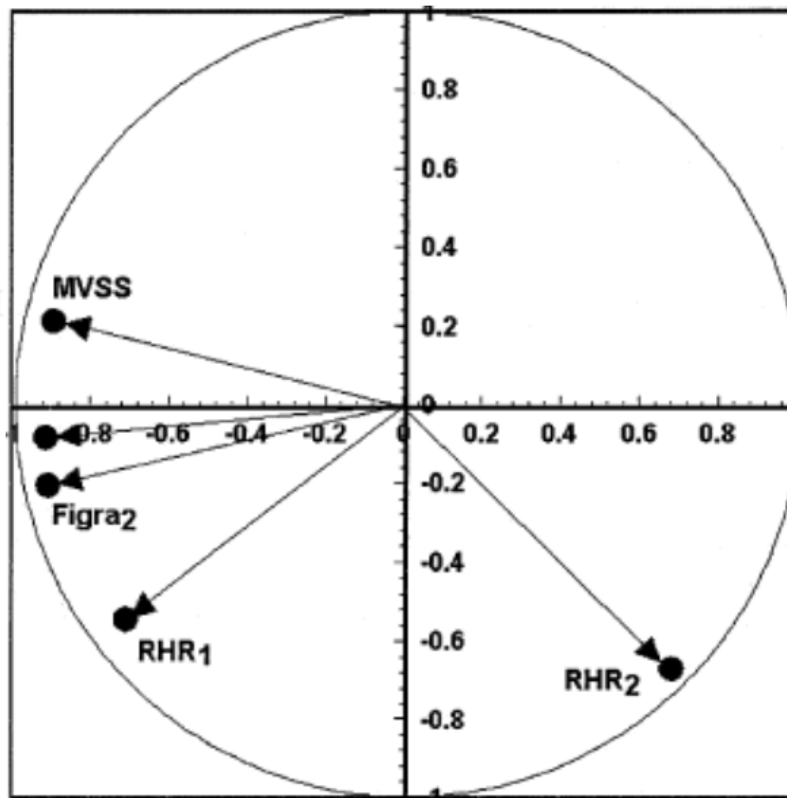


Figure 13. Correlation circle—relationship: cone calorimeter/FMVSS.

From the energy assessment of the foam consumption during 1s, we can find a relation between the propagation speed of the flame and the energy of the tar produced by the combustion (Fig.14).

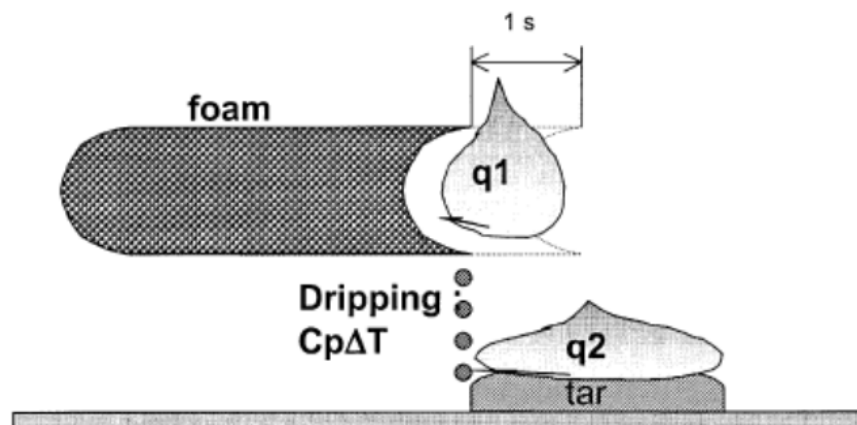


Figure 14. Principle of FMVSS.

As a first hypothesis, we can consider the following relation:

$$q_1 + q_2 - \Delta Q = Q = \text{constant}$$

- ΔQ corresponds to the part of heat used to melt the polymeric matrix leading to dripping.

- q_1 corresponds to the energy released during the first stage of the combustion that leads to the formation of the tar (Figra1).
- q_2 corresponds to the energy released by the combustion of the tar (Figra2).

This relation indicates the different strategies to decrease the value of RHR1 (and so Figra1), that is to say the flame spread in the FMVSS 302 tests:

- To decrease the total heat evolved Q using specific FR additives.
- To decrease the heat released during the first stage of degradation of the foam and as a consequence to decrease the heat fed back to the virgin polymer (decrease in Figra1).
- To increase RHR2, that is to say to reduce the energy of combustion by dripping.
- To delay the heat released by the tar. When the foam is molten, the tar starts to burn and this tar is not immediately lost by dripping. Hence, it is of interest to delay the combustion of this tar to enable it to drip (decrease in Figra2). An increase in RHR2 is not sufficient to reduce the flame spread and it is important that the high energy tar degrades at a later stage.

Hence, we may propose that the flame propagation rate in FMVSS 302 testing is much lower when easy melting and dripping allows heat reduction and tar dripping. It may be proposed that q_2 corresponds in fact to the almost complete combustion of the tar.

Comparing the RHR curves of foams processed with variable water level, we note that the density of the foam strongly influences the first RHR peak (Fig.15). The higher the water content (the lower the density) the faster the step of melting under cone calorimeter conditions.

The effect of density on RHR1 may explain the previous correlation found between density and FMVSS 302. Low density leads to rapid melting and to a high flame propagation rate.

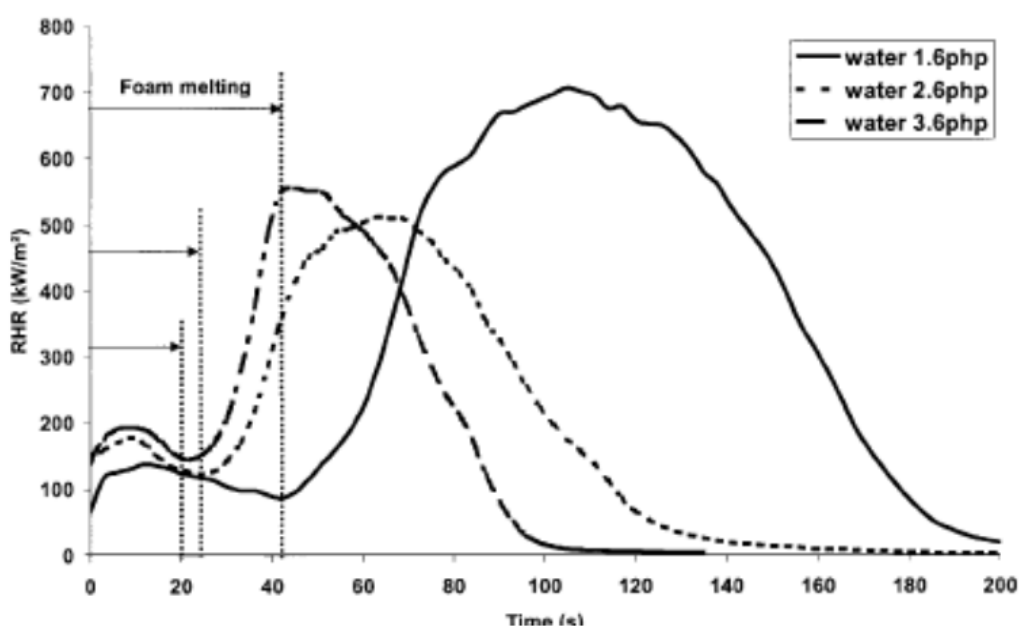


Figure 15. Effect of density on the melting stage of polyurethane during combustion

1.4.2. Cone calorimeter—British Standard

Ignition Source Crib 5 test to SI 1324 Sch. 1 Pt. 1

The statistical computation was made considering the two different sets of foams: the foams containing TMCP–melamine and the ones containing TDCP–melamine. The level of fire retardant additives has been included in the computation but is not shown on the circles of correlation.

Considering the TMCP–melamine foams (Fig.16) it is of interest to note that the lower are Figra1 and Figra2, the lower are the burn times, TWL and DWL. We also note that T_2 is strongly inversely correlated with the data of SI 1324 Sch. 1 Pt. 1, that is to say the higher T_2 the

Better results under the SI 1324 test (lower TWL, DWL and burn time).

The statistical computation of the data from the formulations TDCP–melamine clearly shows that the fire behavior of these foams in the SI 1324 test is linked to the second stage of degradation of the foam in the cone calorimeter (Figra2 and T_2). Indeed, the Figra curve represents the fire growth rate of foam during combustion.

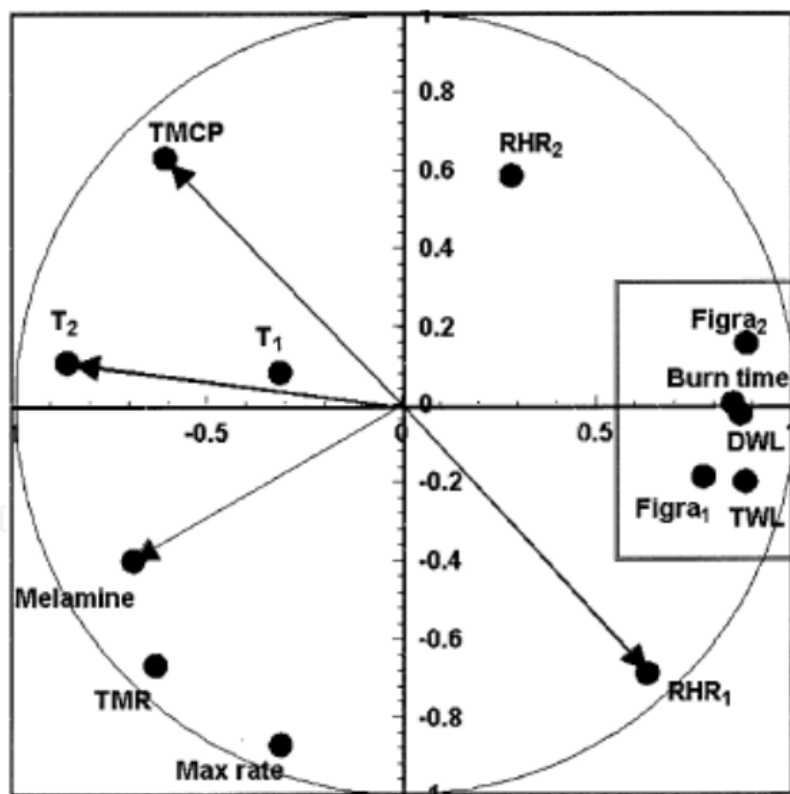


Figure 16. Correlation circle—relationship: cone calorimeter/SI

1324, TMCP–melamine formulations

A high Figra means a high rate of flame propagation and so leads to a high weight loss of the material. Hence, it is not surprising that Figra curves are strongly linked to the BS5852

results. The combustion of PU foam occurs in two steps: the “melting” of the foam and the combustion of the tar. The tar combustion is the most exothermic part of the combustion. A decrease in the heat released by the tar reduces the flame propagation and leads to a decrease in the weight loss of the foam (Fig. 17).

The TDCP and TMCP additives differ in their chlorine and phosphorus content and also in their temperature of degradation. TMCP degrades earlier than TDCP (150 °C and 210 °C, respectively); this temperature corresponds to a 5 wt. % weight loss under thermo gravimetric analysis conditions). A previous study [15] has clearly shown that TMCP is efficient in the early stage of combustion but no interaction with melamine is observed (temperature of 5 wt. % weight loss of melamine is 290 °C). TDCP acts later and when melamine starts to degrade about 50 wt. % of TDCP is available in the system, so a strong TDCP–melamine synergy is observed. The use of TDCP or TMCP in combination or not with melamine leads to very distinctive fire properties of the foams.

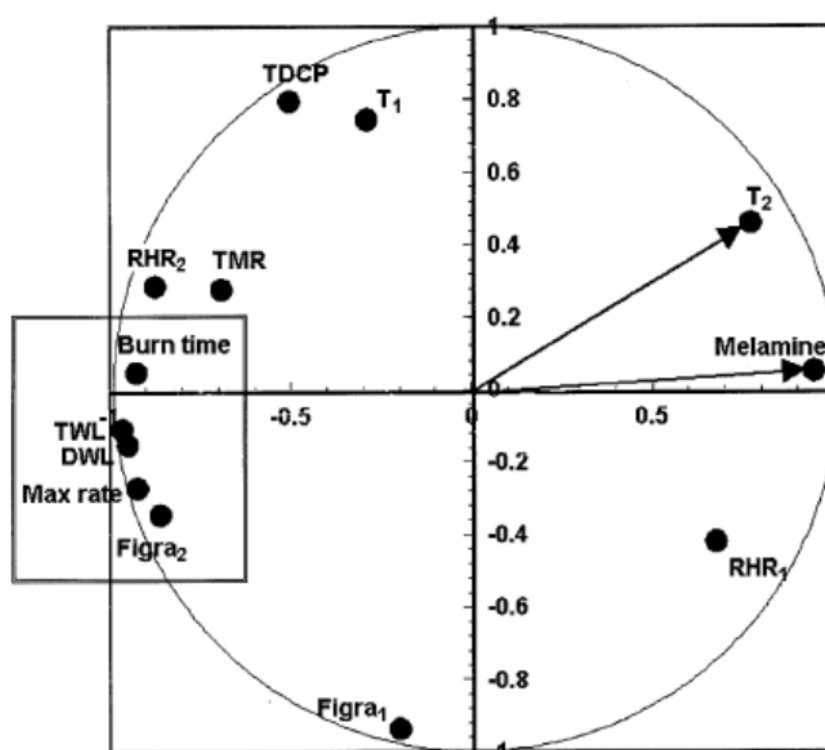


Figure 17. Correlation circle—relationship: cone calorimeter/SI

1324, TDCP–melamine formulations

Considering the TMCP–melamine foams, it is of interest to note that the higher the TMCP content the lower is RHR1. That confirms the early effect of TMCP that acts by decreasing the heat released by the foam in the first stage of the combustion. Secondly, the melamine content is inversely correlated with RHR2. As described previously, the temperature of decomposition of melamine is high (290 °C) and this inverse correlation indicates an efficiency of melamine during the combustion of the tar.

Regarding the TDCP–melamine formulations, we note a positive effect of the TDCP amount on the RHR1 peak. Even if TDCP degrades later than TMCP, a part of the TDCP is efficient in the first stage of the combustion.

The melamine content is strongly correlated with the SI 1324 data. High melamine content leads to a decrease in TWL, DWL burn time and maximum rate of weight loss.

The Fgra2 and RHR2 peaks are also correlated with these data.

1.4.3. Properties–FMVSS 302

The statistical treatment shows that the FMVSS 302 rating is an inverse function of the density of the foam which is itself a function of the water index (Fig. 18). No significant relations may be proposed between FMVSS 302 and porosity or TDI index because data did not show any variation of the porosity (same SnOct content) and only a low variation of the TDI index.

The porosity index of the foam is strongly correlated with the SnOct range used in the foam manufacturing.

The previous study of conventional foams has revealed correlations between the FMVSS 302 testing and these parameters. The PCA study shows the absence of correlation between the EO content, the porosity (and so the SnOct range) and the index of the foam with the FMVSS 302 testing. However, it clearly shows that FMVSS 302 is strongly and inversely correlated with the density of the foam as it has been previously supposed.

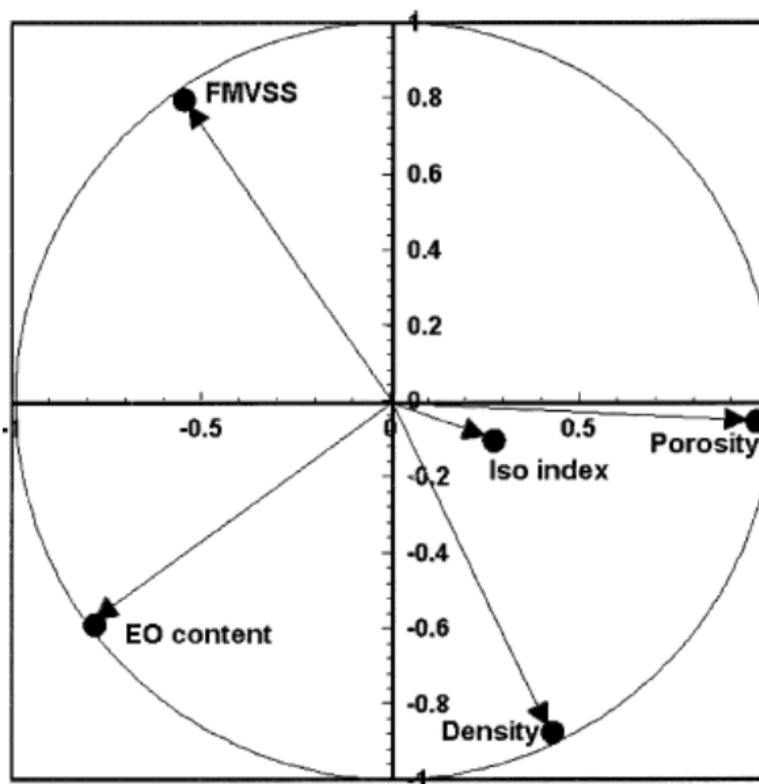


Figure 18. Correlation circle—relationship: physical properties/FMVSS.

Author details

Ahmadreza Gharehbagh

Iran Polyurethane Mfg.Co. NO.30, Tehran, Iran

Zahed Ahmadi

Color and Polymer Research Center, Amirkabir University of Technology, Tehran, Iran

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