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1. History of EPDM Rubber
Development of Rubbers against thermal resistance and engine power

Source Bayer AG

Engine power (kW/liter engine capacity)

Development of C.F. Benz 1886
**History of EPDM-Rubber**

1953  
K. Ziegler invents *titane* catalysts for ethene polymerisation to linear polyethylene  
G. Natta transfers this process later to propene resp. Polypropylene

1963  
Ziegler and Natta receive for their work the Chemistry Nobelprice

1964  
75% of all rubbers become synthetically produced

1964  
Catalyst system of Ziegler-Natta (Z-N) enables the industrial *production of EPM* and  
*EPDM* primarily in USA

1967  
Production of first bales of *EPDM* from DSM in Geleen, NL, called Keltan.  
Plant capacity of 12´000 t/a, 2017 180´000 t/a
History of EPDM Rubber

1968

First EPDM-applications in Europe single ply EPDM roofings in NL

still in service today, just and within the specification

50 years ago start of application of elastic granules (recycled tire, ELT granules)

and later specially formulated EPDM-granules for sports surfaces

In the Olympic Games in Mexico the athletes lined up on synthetic tracks for the first time

The continuous improvement of EPDM-rubbers connected with an extrem increasing of EPDM consumption happened till this day with a lot applications as a result

EPDM applied >70% in the automotive-industry
<table>
<thead>
<tr>
<th>Year</th>
<th>NR</th>
<th>SR</th>
<th>NR/SR</th>
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<td>28.4</td>
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**Rate of growth**

1990-2017: NR 140%, SR 50%, NR/SR 80%
2. What is EPDM chemically?
**EPDM – ETHYLENE PROPYLENE DIENE M**

\[ \text{---}[\text{---CH}_2\text{---CH}_2\text{---}]_m\text{---}[\text{---CH}_2\text{---CH}---]_n\text{---}[\text{---Diene---}]_o\text{---} \]

1

\[ \text{CH}_3 \]

– **EPDM** is a **terpolymer** of Ethylene, Propylene and a non conjugated Diene

– The diene is usually dicyclopentadiene, ethylidene norbornene, vinyl norbornene

– **M** stands for a saturated poly**M**ethylene hydrocarbon main chain (M-class refers to ASTM D-1418, rubbers having a saturated chain of the polymethylene type)

– **EPDM** has a ratio of ethylene/propylene between **85/15%** and **45/55%** by weight
The mainly used dienes in EPDM:

- 5-Ethylidene -2-norbornene (ENB)
- 5-vinyl-2-norbornene (VNB)
- Dicyclopentadiene (DCPD)

Diene content may vary from 0,9% up to 12% by weight of the composition. Higher diene percentage enables faster vulcanisation and higher crosslinking degree.
One double bond -1- of the diene reacts with the main chain, the second -2- remains for a sulphur vulcanisation.
EPDM chemical

• EPDM is a nonpolar, fully saturated polymer, with a very low level of unsaturation in the side chain of the polymer

• EPDM is a real synthetic rubber (SR), one of more than 40, at this time known, different rubber groups

• EPDM is not compatible with polar compounds, it can be vulcanised with sulphur curatives as well as with peroxides, then with a higher curing efficiency

• EPDM are the third-largest volume of SR, consumed worldwide, after SBR and BR (latter are mainly used for passenger tires)
3. EPDM Production
Synthetic Rubber Production
From Mineraloil and –gas to Synthetic Rubber

**Feedstocks**
- Mineraloil/-gas
- Hydrocarbon-condensates
- Naphta
- Butane
- Propane
- Ethane

**Basic products**
- Benzol
- Ethylene
- Rawbutadiene
- Propylene
- Ethylene
- Rawisobutylene
- Rawisoprene

**Key precursor**
- Styrene
- Ref. Butadiene
- Acrylnitrile
- Ref. Isobutylene
- Ref. Isoprene
- Ref. Diene

**Final product Rubber**
- Styrene butadiene rubber SBR
- Butadiene-rubber BR
- Acrylnitrile-Butadiene rubber NBR
- Ethylene propylene rubber EPM/EPDM
- Isoprene, Butyle and Halobutyle rubber IR, IIR,CIIR,BIIR
Production of EPDM Rubber

➢ Three different processes

• **Solution** process most widely used
• **Suspension** process enables lower solvent
• **Gas phase** process no longer used

➢ Catalysts

• **Ziegler- Natta (Z-N)** metall organic compound, vanadium based
• **Metallocene** group of metallorganic compounds, where one metall- atome is between two organic ligands like a sandwich
• **ACE- Advanced Catalyst EPDM** new catalyst system, special titane complex
Production Scheme for EPM/EPDM Rubber Solution-Process

Ethylene
Propylene
Diene
diluted in solvents

Source: LANXESS „BUNA EP“
50 years of advancements through continuous improvement of the...

- process
- catalysis
- ecology
- application technology
- economy
- product

In the past gel-formation was a big problem in the production-process. Only the ACE-technology (Advanced Catalyst EPDM) this problem could be solved. However oxidative gels can still be formed during storing and compound mixing in the rubber plants.
Major global EPDM-rubber producer:

- Arlanxeo > 20% market share, the leader!
- ExxonMobil
- DOW Elastomers
- SK Chemical
- Sumitomo
- Mitsui Chemicals
- PetroChina
- JSR Corporation
- Kumho Poychem
- ENI
- and others
4. EPDM properties and applications
EPDM Rubber Properties

➢ A huge number of EPDM-rubbers enables a use oriented compouding

➢ The different EPDM polymers may vary by...

  – molecular weight (MW)
  – MW-distribution (MWD)
  – viscosity
  – content of polyethylene and polypropylene
  – amount of diene
This table shows **26 different EPDM rubber**, only from one producer the **ExxonMobil**
e.g. Arlanxeo offers more than **50 EPDM polymers**

### Typical properties

<table>
<thead>
<tr>
<th>Grade</th>
<th>Oil phr</th>
<th>Mooney viscosity ML ((1+4)) at 125°C</th>
<th>Ethylene weight % ASTM D1646</th>
<th>ENB weight % ASTM D6347</th>
<th>MWD type</th>
<th>Form</th>
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<tbody>
<tr>
<td>404</td>
<td>-</td>
<td>28</td>
<td>45</td>
<td>-</td>
<td>Very broad</td>
<td>Dense bale</td>
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<tr>
<td>703</td>
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<td>21</td>
<td>72</td>
<td>-</td>
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<td>Bale</td>
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<tr>
<td>706</td>
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</table>

**Terpolymers - low to medium diene**

<table>
<thead>
<tr>
<th>Grade</th>
<th>Oil phr</th>
<th>Ethylene weight % ASTM D1646</th>
<th>ENB weight % ASTM D6347</th>
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<td>67</td>
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**Terpolymers - high diene**

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<td>73</td>
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</tr>
</tbody>
</table>

1. ML employed at 125°C

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**Mooney viscosity:** 17-115  
**Ethylene weight %:** 45-78  
**ENB weight %:** 0.9-10.0  
**MWD:** narrow up to very broad and bimodal
EPDM Rubber Properties

- High filler and plastisizer loading
- Lowest density of all commercially available rubbers (0.86 g/cm³)
- Fast mixing, no mastication necessary
- Big versatility in processing properties
- Delivery form are bales and crumb
EPDM Elastomer Properties

• Very good resistance to aging (heat and oxidation)

• Excellent weathering -, ozone-, UV light-, and oxigen resistance

• **Chemical resistance** in polar fluids as alcohols, ketones, glycols, diluted acids and alkalines – unstable in mineraloils and all unpolar fluids

• **Outstanding water and steam resistance**

• **Flame retardent** - if particulary formulated with high loadings of Al- or Mg-hydrates

• **Good low temperature properties** – dependent on the ethene content >65% worse
EPDM Elastomer Properties

- Hardness: 30 Shore A up to 50 Shore D
- Tensile strength: 7 - 20 Mpa
- Elongation at break: 150 – 700 %
- Heat resistance: up to + 150°C
- Low temperature: up to – 40°C
- Ozone and weather: excellent
- UV resistance: black materials excellent, for colored EPDM only with UV-absorber good
- Chemical resistance: excellent, best from all unpolar elastomers, exception polar fluids unstable

➢ all properties vary with the MW and the ethene content, as well as with the composition of the elastomer compound (filler- and plastisizer amount and the crosslinking degree)
Application of EPDM Elastomers

The big versatility of EPDM elastomers open a great field of different applications in various industries:

➢ **Automotive**

The most important sector, >70% of elastomers are used in automotive engineering.

➢ **Building and Construction**

A lot of applications, particular different sealings, this sector belongs also to: granules for rubberized surfaces, like athletic tracks, playgrounds, tennis courts and infill in artificial grass.

➢ **Further Applications**

such as cables, consumer goods, drive and transmission belts, fire hoses, sealings for washing machines, printing and paper rollers, shoe soles, key pads…. and a lot others.
5. Compounding, Mixing, Vulcanisation
Compounding

➢ Three main objectives have to be respected:

• **Requirements** (of the final product)

• **Processability** (mixing and vulcanisation)

• **Profitability** (cost-benefit ratio)

➢ **Rubber compounds** are composed of minimum 2 up to maximal 20 different ingredients with a big dependence of the chemist, perhaps an overkill!
Requirements for EPDM Granules

- Outstanding long term weathering resistance
- UV resistance in practice and lab-tests (colour stability)
- Mechanical properties corresponding with practical function
- Compatible with PU- coatings and binders
- Environmentally compatible
- Consistant colour for different batches
- Colour consistance after longterm outdoor use
- Low odour, Low dust
- Conform with relevant standards
- High cost-benefit ratio
All requirements must be considered for the tailored, coloured elastomer granules:

- Longterm weather resistance
- UV resistance
- Standards e.g. EN 14877,15330-1
- Colour stability and constancy
- Environmental properties
- Mechanical properties
- Flame retardent
- PU compatible
- Low odour, low dust
- High cost benefit ratio

The tailored elastomer for coloured granules??
only EPDM based elastomers
are predestinated in sport surfaces applications!!!
EPDM RUBBER

FILLER

PIGMENT

Crosslinking system

Plastisizer

Stabilizer

MIXING

Rubber Compound

ISSS Technical Conference 2018-10-25/26, Cagliari

Günter Preisser 33
Mixing

➢ Discontinuous mixing

• Weighing
  – automatically
  – manually

• Mixing cycles

1. **Crushing** and masticating of raw rubber (bales of 20 - 25 kg)
2. **Increasing** of the polymer surface
3. **Incorporation** of ingredients (filler, plastisizers, chemicals)
4. **Degradation** and **dispersion** of filler agglomerates
5. **Distribution** (filler reduction in the polymer matrix)
6. **Discharging** on a mill or extruder
intermeshing rotors
rotor speed 30-90 turn/minute
knead blade (rotors)
back gauge
techical goods-
Volume 30-250 l
tire-production up to 700 l
back gauge discharging

filling shaft
filling flap
ram
cooling
mixing chamber

Internal mixer
Mixing

➢ Continuous mixing

• *Twin screw mixing* in modular co-rotating twin screw extruders

  - The *ingredients* must be primarily *separately balanced, premixed* and then charged into the mixer, plastisizer and crosslinking system mainly separated

  - *Vulcanisation* also *continuous* in hot air or steam, temperature > 160°C

  - The *granulation* occurs after the vulcanisation in the *same processline*
Rubber Compound

Forming, shaping + Temperature 150 - 200°C

Vulcanisation

Crosslinking

ELASTOMER
6. Standards for sportsurface granules
EPDM Granules Standards

Unfortunatelybesideinternationalstandards(e.g.ENandISOstandards)arestill
nationalstandardsexisting. ThemostcommonstandardsinWesternEurope:

• EN 14877 Synthetic surfaces for outdoor sport areas
• EN 15330-1 Synthetic turf and needle-punched surfaces primarily for outdoor use
  Part 1 Specification for synthetic turf surfaces for football, hockey, rugby union training, tennis and multi-sports use
• DIN 18035-6 Sport grounds – Part 6: synthetic surfaces
• DIN 18035-7 Sport grounds - Part 7: synthetic turf areas
EPDM Granules Standards

• **Requirements** of relevant standards are partially **difficult to fulfill**

• **Particular** the very low **zinc-content** in DIN 18035-6 of **< 1,0 mg/l**
  in DIN 18035-7 **< 0,5 mg/l**

• The **compliance with regulation** could activate an undervulcanisation

• **Standards for EPDM elastomers** for food- or potable water-applications
  allow higher **zinc content**

• **Tests** have to be checked **on testslabs** and **on granules**
  to make sure that testplates and granules respect an **equal material**
EPDM Granules Standards

• All effort has to be made to get harmonized with international standards, like ISO or EN, and consequently all national standards should be eliminated.

• What are the experts, your knowledge with the current standards, are improvements or revising necessary?

• Please take the chemists from the EPDM-elastomer producers with you into the standardization boat!!

Standard should always respect satisfiable properties only as good as necessary!
7. Properties EPDM Granules state-of-the-art
Comparison with other granule materials
EPDM Granules state-of-the-art

- **Granule Size**
  narrow particle distribution, beside turf and crumb

- **Environmental**
  low heavy metal content, less or no VOC, NOA, PAH

- **Colour stability**
  UV resistant pigments

- **Low dust content**
  improved technology in granulation process

- **Noise absorption**
  use of special fillers, high filler loading

- **UV resistance**
  UV absorber, more practice adapted UV-tests up to 8000 hours, in UV-A and -B

- **Flame retardant**
  EN 13501-1 - without halogen- and phosphor compounds

- **Mech. Properties**
  abrasion and elasticity at the application range
<table>
<thead>
<tr>
<th>Material</th>
<th>Weather/temperature resistance</th>
<th>UV/ozone resistance</th>
<th>Mechanical properties</th>
<th>PU-compatibility</th>
<th>Environmental compatibility</th>
<th>Odour</th>
<th>Costs</th>
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<td>EPDM coloured granules</td>
<td>++</td>
<td>UV + O₃ ++</td>
<td>+/-</td>
<td>+</td>
<td>++</td>
<td>+</td>
<td>+/-</td>
</tr>
<tr>
<td>recycled black granules (ELT-end of life tyres)</td>
<td>+/-</td>
<td>UV + O₃ -</td>
<td>+</td>
<td>+/-</td>
<td>+/-</td>
<td>-</td>
<td>++</td>
</tr>
<tr>
<td>TPE-V coloured granules</td>
<td>+/-</td>
<td>++</td>
<td>+/-</td>
<td>++</td>
<td>++</td>
<td>++</td>
<td>-</td>
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<tr>
<td>Cork-coconut fibres</td>
<td>+/-</td>
<td>+</td>
<td>--</td>
<td>+</td>
<td>+?</td>
<td>???</td>
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</tbody>
</table>

**Comparison of properties**
8. EPDM Granules Cause of Failures in the Past
Cause of Granules Failures

➢ Main failures

- **chemical degradation** (sticky surface, no longer usable soft granules)
- **mechanical degradation** (destroyed granules)
- colour change
- chalking
- blooming
Cause of Granule Failures

➢ Main causes

- Insufficient degree of crosslinking
  not fully vulcanised, or reversion due to too high vulc.-temperature

- UV absorber
  inhibited from crosslinking system and not reactive, general false UV-absorber

- Gel content

- Diene content too low
  reduced crosslinking degree

- Peroxide crosslinking
  residual of decomposition products and inhibition of UV-absorber

A lot of possibilities could be responsible for a degradation, only with phsyico-chemical analytics the root cause can be evaluated!
Cause of Granule Failures

• Further possible causes:

  – Heavy metals influence especial Cobalt avoiding
  – Chlorined mains water
  – Cure temperature not correct (too low or too high)
  – Residual from the catalysis system
  – Pigment formulation anorganic pigments e.g. Ironoxide, titandioxid

A lot of possibilities could be responsible for a degradation, only with phsyico-chemical analytic the root cause can be evaluated!
Influence Factors on EPDM Degradation

➢ How can a degradation be avoided?

- **UV light**
  - use of particular UV-absorber

- **High outdoor temperature**
  - quenching with cold water, high amount of white fillers

- **Residual of catalysts**
  - dependence on the polymer processing use of purer polymers

- **Crosslinking system**
  - avoiding of peroxide curing and general not any undervulcanisation

Realise: chemical reactions are faster with UV-light and temperature equal for degradations
9. Future of Granules in Sports Fields
Future of Granules in Sports Fields

➢ Which changes can be expected?

• EPDM- sizes granules replaced by turf, stripes or other shape
• EPDM-granules with a self gliding surface
• EPDM-granules spongy, foamed, waterabsorbing
• EPDM granules bio-based EPDM combined with bio based ingredients
• EPDM/TPE and –TPV blends with improved properties
• TPE/ TPV with better thermostability
• TPE/ TPV more price competitive with EPDM

Nowadays EPDM is the optimal choice for coloured granules
Future of EPDM Granules in Sports Fields

• Further innovations in the catalyst-systems and the polymerisation process could lead to further very interesting new polymers with use oriented properties

• A rethinking of the rubber producers, away from the absolute dependency on fossil fuels to more renewable resources

• Using of the world’s first bio-based EPDM rubber, production start was 2017 in Triunfo (Brazil), commercialized by ARLANXEO under the trade name Keltan Eco

   Keltan Eco is produced by an solution process with an improved Z-N catalyst technology. The bio based ethylene originates from sugar cane, converted to ethanol and dehydrated to ethylene
Future of EPDM Granules in Sports fields

Isn´t a biobased EPDM a big challenge for EPDM-granules producer?

Using a green polymer, based on renewable resources, in a green outdoor area, like sports fields?

What a benefit announcing granules based on green polymers with

- A green non mineraloil plastisizer, a vegetable oil
- Fillers and pigments only from natural sources
- An environmental compatible crosslinking system

Biobased EPDM, with ingredients from natural sources, could change the bad reputations, when media discredit EPDM granules as harmful. What a future prospect and a guaranty for the survival of EPDM granules in sports fields for the next, how many ??? decades
Thank you for your attention