SYNTHESIS REPORT

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PROJECT COORDINATOR : Henkel KGaA

PARTNERS : NKT Research Center
Grecian Magnesite
Ecole Nationale Superieur de Chimie de Lille

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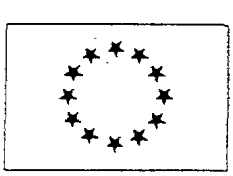

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ILFPO

Ion Linked Flame retardant POlymers

Names and addresses of the coordinator and of partners

1. Henkel KGaA, W. Klauck, R. Schelbach, D-40191 Dusseldorf, Germany
2. NKT Research Center, Bjørn Marcher, Sognevej II, DK-2605 Brøndby, Denmark
3. Grecian Magnesite SA, L. Athanasakis, 45. Michalacoparlou STR, 115, 28 Athens, Greece
4. Ecole Nationale Superieur de Chimie de Line, R. Delobel, B.P. 108, 59652 Villeneuve d' Ascq Cedex, France

Abstract

In general, it can be said, that the work between the partners has become more independent as was foreseen at the beginning of the project. All the partners worked on the subject to use renewable raw materials for different applications.

Grecian Magnesite prepared Magnesium based fillers for polymercompounds and as reactive salt in ionic polymerization processes. There Grecian Magnesite was very successful and developed both a laboratory and a pilot plant scale process for converting MgO from its own mine in Greece to $Mg(OH)_2$, flame retardant grade.

Henkel developed a new adhesive which is based on oleochemical oligomers described in the abbreviations of the samples in the annex. The adhesive fulfils market requirements for paper glues and in formulations with some percentages of petrochemical based polymers for high performance products. Project result led to a new R&D-project for oleochemical modifications of calciumcarbonates for DIY products.

The NKT-Research center investigated and produced a halogen free polymer for cable insulation based on EVA which fulfils all technical requirements and market needs. The product is very interesting because of the price which is low compared with the best product currently available in the market.

The University of Lille provides new knowledge and better understanding of pyrolysis reactions and polymer behavior within polymerblends. Characterisation of complex polymer compounds is a difficult subject and needs highly experienced personnel.

Based on this general overview, we like to describe the different tasks on which the partners worked on. The results should give an idea of all the subjects mentioned. in

the work program. The partnership was excellent and all of the partners agreed that a fruitful work atmosphere was present throughout the whole project. In addition the work was done very productively all the time.

Work done on the individual tasks:

Task 1: Preparation of COOH-terminated oleochemical oligomers

Different renewable raw material based on oligomers as described in the annex were produced. Referring to the work program 26 samples (Soy bean-, Linseed-, Castoroil, Sojapolyol and Dimeric fatty acid), 1kg each, were prepared and delivered to the partners NKT and E. N.S. C. L.. They were characterized by S2 = No of acid g-maps, OHZ = N" of OH-groups, VZ = N^{O} of ester groups and JZ = N^{0} of unsaturate groups.

Task 2: Basic formulations

Based on the samples available at Grecian Magnesite and commercially used metal salts 20 samples of different basic compounds were produced. Different metal salt (MgO, $Mg(OH)_2$, CaO, $Ca(OH)_2$, ZnO, $Zn(OH)_2$...) were used with a variation of molar ratios. Depending on the salt content the product viscosity increases with the metal salt content. Other carboxyl-terminated oleochemicals like soybean oil polyol and castor oil terminated with phthalic anhydride were obtained as iess reactive than the pure soybean oil/dimeric fatty acid.

To improve the mechanical properties for application as chlorine free cable insulation, samples from task 2 were modified' with conventional polymers and resins. Products with 10% up to 40% EVA-copolymers show an encouragingly improved property. An interesting polymer mixture can also be a polyamide based on dimeric fatty acid (Macromelt MM 607 i) which is based on oleochemical raw materials.

Ring-open products of epoxy dized fatty acid methylester reacted at room temperature to, a solid material. By adding surplus molar ratio of metal salt generally solid products were obtained. The most reactive salts were MgO (Maglite A) and $Ca(OH)_2$.

A variety of MgO-samples of Grecian Magnesite have been tested. Only sample 74/8 (see final repost of Grecian Magnesite) reacted as well as commercially available Maglite A. The most interesting samples (74/8 and 74/4) will be further investigated in an effort to optimize their performance depending mainly on grain size distribution and crystallinity. It is worth mentioning that samples with very fine particle size seem to lose their reactivity, a trend which is possibly due to increasing agglomeration.

Based on the 27 samples prepared, Henkel tested three further interesting substances as MSA adduct. Soybeanoil, rapeoil and sunfloweroil were converted by a molar ratio of 1 part oil to 3 parts MSA. Combining these new oleochemicals with metal salt, no reaction takes place. But adding a small percentage of water (app. 2%) a potlife of 4-10 minutes was obtained. This behavior reminds one of a 2-component product. Special applications for under water coatings might be possible. However, the film properties need to be improved since the coatings are to hard and brittle:
These results led to additional work for testing basic formulations as given in the updated bar-chart diagram.

Task 3: Testing of basis formulations

Standard testings on shear-strength were made by using standard beech-plywood test-samples (size 8x2,5x0,5 mm). Samples 1, 2, 7, 11, 12 and 17 (mixed with 10 % Maglite A and 10 % glycerin) fulfil the requested goal of $>2\,N/mm^2$. However, shear-strength is independent of the acid number. Mixtures of sample 1 and different bivalent metal salts reacted to products with low shear-strength.

The highly filled polymer matrix was improved by adding a modified maleic anhydrid polymer. Silane, treated with hydroxides, EVA and an oleochemical oligomer compound fulfil the requirements in tensil strength (10 MPa) and elongation at break (150 %). Further the LOI (limiting oxygen index) measured according to ASTM standard (D-2863) complies with 36% the goal of the project, too.

Several samples were mixed with 10% Maglite A and standard film pieces (5 x 5 x 0,3 cm) were formed. These samples were stored in water at different pi-i-values (2,7 and 11). As expected. samples with high content of polaric groups showed a bad water uptake resistance. Samples with long unpolar hydrocarbon chains had a good resistance against water.

Task 3.2: Spectroscopical characterization

In order to get reference data, combinations of fire retardant fillers and polymers (in particular EVA - copolymer ethylene-vinyl acetate) have been investigated by four parameters: ignition time, rate heat release (RHR) maximum value, heat evolved and effective heat of combustion.

The chlorine free model compounds of NKT present an acceptable performance in comparison with the best commercially available references.

For less specific oils and fats supplied by Henkel systematical spectroscopical characterization was done (IR, ^{13}C- and ^{1}H NMR). These data for oligomers as well as for polymers helped to understand the reaction mechanism.

Task 4: Investigation on influence of fillers and additives

The initial' goal was to prepare reference compounds with properties as good as possible. Considering that, NKT optimized compounding conditions for parameter temperature, blending tirneand shea.rrate with existing materials.

After optimization of parameter conditions, well known ATH (aluminum trihydrate) filler was used as a reference for $Mg(OH)_2$ filled polymers.

The results obtained were very encouraging. We expect to improve application behavior after optimizing this compounds. In order to get better results for surface treatment of the filler by fatty acid esters a twin screw extruder will be used instead of a Barbener type compounder.

During this first step in developing magnesium hydroxide the results in terms of thermal decomposition and flammability resistance were encouraging, too. The flame retardant properties of $Mg(OH)_2$ as" a filler were tested by filling EVA. A dramatically decrease of RHR of about 80% was obtained. THE decreased also by approximately 45 %. An on-line FT-IR analysis of gaseous effluents shows a good correlation with literature data.

The most reactive magnesiumsalt is MgO (Maglite A). Until now, it is not clear, what the major parameter is that most significantly influences the reactivity of MgO samples. Crystallite size and specific surface area should play an important role. To identify the most important factor is the key factor for optimization of existing MgO products. Furthermore, Grecian Magnesite developed and delivered $Mg(OH)_2$ samples to NKT anticipated to be suitable for use as flame retardant fillers in plastics. These samples reached the criteria for the first mid-term-assessment point, i.e. purity >85 % a'nd the particle size distribution (more than 20 % in the range 1 - 20 mm). In addition, Grecian Magnesite had the opportunity to produce $Mg(OH)_2$ and provide it to ILFPO.

Investigations to deeply understand magnesium based fillers needed more time than estimated. Due to the importance of this task, all partners agreed at the last annual meeting to continue the work. This prolongation did not influence the project costs but led to a closer cooperation between Grecian Magnesite and NKT-research center which was in this depth not part of the project application and work program.

Task 4.2: Polymers with high filler acceptance

The present highly filled polymers reach "the mid-term assessment criteria. Their elongation at break is 187% (criterium $>$ 150%) and tensil strength was obtained by 13 MPa (criterium $>$ 10 MPa). This flame retardancy, characterized by the limiting oxygen index (L. 1.0.) is good with the 36% obtained (criterium $>$ 28 %).

The best sample with regard to the share strength reaches 4 N/mm^2. This value fulfils the mid-term assessment criterium of > 2 N/mm^2.

Task 5: Optimization of basic formulations

Grecian Magnesite submitted 8 samples of MgO and $Mg(OH)_2$ to Henkel. At Henkel they had been converted with samples 1 and 7 to compare the reactivity with the commercially available product Maglite A. The highly specific surface area influenced the reactivity positively. For polymerization applications MgO grades should have a surface area in excess of 150 m^2/g and a size distribution of dg0 < 10 microns.,

$Mg(OH)_2$ as a filler for polymer compounds tends to agglomate. This agglomeration does not allow a homogenous dispersion in the polymer matrix to improve tensil strength and elongation at the break of polymer compounds. Further investigations on hydration process of $Mg(OH)_2$ have been started.

Based on tasks one and two, Henkel prepared some new (and not planned) oligomers based on vegetable oil and MSA at high temperatures. DieIs-Alder- or En-reactions can take place at the double bonds. These MSA adducts do not react with metal salt. However, by adding' water to the mixture the hardening-process takes only a few minutes to some hours (depending on water content). Based on these results further tests to use this compounds as a water-curing adhesive have been started and should be reported at the next meeting.

Grecian Magnesite prepared MgO samples by thermal decomposition of $Mg(OH)_2$. Depending on the temperature a high specific surface area (SSA [m^2/g]) was obtained. At 450°C a surface area of 242 m^2/g was measured. Coinpared with Maglite A (the most reactive commercially available product with 206 m^2/g) reactivity should be higher. A high specific surface area influences the reactivity positively.

Henkel tested two optimized samples from Grecian Magnesite (DHYDMAG 2, DMGHYD 4). Reactions with oligomer Sample i and 7 of task 1 and DHYDMAG 2 shows nearly the same reactivity as Maglite A. Is was the most reactive magnesium oxide from Grecian Magnetite.

NKT tested $Mg(OH)_2$ samples (MGHYD 1 1 and 12) from Grecian Magnesite. Significant improvements in elongation at break and tensil strength took place. For up-scaling trials, Grecian Magnesite produced an amount of 2 kg from the optimized $Mg(OH)_2$ samples. Generally, magnesium hydroxide is, improved a lot and reaches in some areas standards of commercially available fillers.

Task 6: Ecotoxicological testings of the new polymers

In the first approach, ecotoxicological data were available for most of the starting materials. Monomer components like fattyacid methylester, epoxidized fatty acids or diethylenglycole have a good biodegradability and a low toxicity. In general, toxicity is not influenced significantly by polymerizing these monomers. Increasing molecular weight led to a lower biodegradability due to increasing insolubility. However, toxicity of the oligomers is lower than of the monomers (see Henkel page 13-15).

Task 7: Application related tests of prototype compounds

Adhesives:

To improve adhesive properties fat based raw-materials were mixed with different polymers at 130° - 160"C. As a result extremely sticky compounds were obtained by using commercially available polymer contents. A modification of sample *2*, Ca(OH) and a dimer fatty acid led to very interesting materials in the area of application as hotmelt adhesives.

To develop a suitable and commonly used application form for the modified basic formulations the compounds were emulsified in water. These dispersions are forming transparent and sticky films with shear-strength of $1.9\,N/mm^2$. Results of the pre-screening phase encourage us to put more effort into converting fat based raw-materials with metal salts into dispersions for testing their adhesive properties.

Different ageing tests for UV-stability, heat- and oil-resistance were undertaken. UV-stability is in range of the required limits, the mechanical properties have not been significantly changed. All tests are based on the ASTM G 53-88 standard. The resistance against heat and oil is (as far as the results are present) encouraging. Only tensil strength for the tested compounds changed by +21.4 % which is 1.4 % above the limit.

Cable Sheating

Commercially available base resins and magnesium hydroxides were used to produce a prototype compound for cable sheathing. The major aim was to produce flame retardant and chlorine free compounds to substitute PVC-based polymers. For being successful and developing marketable products not only renewable raw materials from Henkel were applied- The new halogen free formulations are mainly in accordance with product requirements (see page 2 of NKT report). Ageing and stabilit y properties need improvements.

Reference testings with the best commercially available compounds show a R. H. R. maximum of about 175 kW/m^2. The six best already tested prototype compounds of NKT give a maximum of 150 to 170 kW/m^2 which is even' better than the best reference. As the limiting oxygen index (L.I.O.) is widely used in the cable market, some additional measurements on the prototype compounds were made. For our mid-term assessment point we want to be $>28\%$ L.O. 1. Most of the samples reach

$>35\%$ **and** more. Therefore, this result is in good accordance with the fire performance index (F. P. I.) given in table 4 of E. N. S.C.L. report.

Task 8: Process scale-up

The extrusion of a prototype formulation over an insulated cable gave very good , and satisfactory results.
Magnesium hydroxide in course of development at Grecian Magnesite Research Center is improving in quality. However, the agglomeration problem seems to be the main barrier for this filler to be evaluated at optimized conditions and in a larger spectum of polymer resins.

Task. 9: Usability investigations for further applications

Most of the basic formulations were investigated as solid state materials. Especially for adhesive applications dispersions are of interest. To increase the chances to use project results in current products, Henkel tries to prepare dispersions based on the basic formulations. Shear strength of PUR-modified dispersions were obtained in a range of 5,0 to 9,0 N/mm^2 (measured on beech plywood instruments). This is significantly higher than our requested shear strength of $>2 N/mm^2$. It seems possible to use the dispersions as adhesives, sealants or coating materials. Special application tests will be done in the next months.

The second midterm assessment point criteria were:

a. Ecotoxicological testings of the new polymers and no toxic gases by combustion
b. Application related testings of prototype compounds have to be fulfilled
c. Product cost should be in the range of 2-3 ECU/kg
d. All physico-chemical data given in first assessment point have to be fulfilled

a: The monomers used are commercially available and well-characterized products. In general, all starting materials are of good biodegradability and show a low toxicity (fish-acute toxicity LC 50 1000-10.000 mg/l).
The polymerized products are less biodegradable due to their low volubility. But that is what we expected- Based on the project aim given in the workprogram the assessment point is fulfilled. It was not our goal to prepare biodegradable compounds.

b: Grecian Magnesite has produced 100 kg $Mg(OH)_2$ flame retardant grade on a pilot scale reactor and has delivered samples to NKT for evaluation- Basic formulations of fattyacid methylester and metal salt can be produced in batch-reactors- Pilot scales up to 50 kg for dispersion applications were done successfully at Henkel. No difficulties are expected when scaling up to 200 k g .

c: Product cost for basic formulations are in a range of 2 ECU/kg. Flame retardant polymers based on EVA-copolymers show good product properties. Their price is below commercially available products and additionally the insulation is halogen free.

d: Currently reached physico-chemical characteristics:
- Elongation at break: 178 %
- Oxygen index: 34 %
- Tensil strength: 13 MPa
- Filler acceptance: 61,5 %
- Particle size: 90 % under 7 μm
- Purity: 93 %

Introduction

Many of the currently used plastics contain varying amounts of halogen. One of the major examples is PVC. In case of combustion PVC is problematic because of the formation of toxic and acid gases as 'well as for smoke generation at pyrolysis temperatures.
The present polymers/plastics are usually based on petrochemical products. The negative environmental impact of these materials is well known.

The main objectives of the proposed project are

- to' develop new low cost polymers based on renewable raw materials using oleochemical materials
- to develop specific flame retardant additives and fillers for these new polymere compounds
- to combine these materials with reduced amounts of standard base polymers in order to obtain environmentally compatible, flame retardant polymer compounds with good mechanical properties

About 50 % of the currently used plastics have to fulfil the specification of flame retardancy.
Subsequent commercial development will bring these new materials to direct application within the core business areas of Henkel and NKT, directed towards adhesives, sealants, coatings and thermoplastics for electrical applications.

Technical description

The development of the new polymers mentioned above has been divided into 8 tasks which were performed successively:

Task 1: Preparation of oleochemical oligomers

Task 2: Basic formulations

Task 3: Testing of basic formulations
Task 3. 1: Adhesive behaviour
Task 3.4: Wateruptake

Task 5: Modification of selected basic formulations

Task 6: Ecotoxicological testings of oleochemical oligomers

Task 7: Preparation of dispersions based on basic formulations

Task 8: Process scale up

Results:

The most important aim of the project was the development of halogen-free polymer compounds for various applications, eg., adhesives, cab le insulation and sealants. Commonly used PVC-Polymers are problematic because of its toxic and acid gas formation at pyrolysis temperature. Fire statistics show that 69% of all deaths are associated with post-flashover fires, with the vast majority of deaths occurring outside the room of fire origin. One conclusion is that people die far away from a fire due to toxic gas inhalation. [n addition to the polymer matrix the inorganic filler highly influences flame retardancy. The most commonly used inorganic flame retardant fillers are $Al(OH)_3$ and $Mg(OH)_2$.

Criteria for products to be up-scaled were:

Target products:

Flame retardant halogen free polymers filled with $Mg(OH)_2$ (by NKT)

Adhesives and sealants based on oleochemical raw materials, hence environment friendly (by Henkel)

Flame retardant grade $Mg(OH)_2$ and MgO suitable for polymerization applications (by Grecian Magnesite)

Target product characteristics:

$Mg(OH)_2$ from particle size of 1-20 Micom. and more than 20% in that range $Mg(OH)_2$ purity $> 85\%$.

Storage stability about 2 months at 40 °C

Share strength > 2 Nm/m^2.

Filler acceptance > 6 0 %
Tensile strength > 10 MPa
Limiting Oxygen index > 2 8 %
Elongation on break > 150%
Ecotoxicological testings no toxic gases by combustion
Product cost 2-3 ECU/kg

Reached product performance

$Mg(OH)_2$ from particle size of 90% < 5 μm
$Mg(OH)_2$ purity app. 95%.
Storage stability about 2 months at 40 "C
Share strength 2.7 -5.2 Nm/m^2
Filler acceptance 60 %
Tensile strength > 12.6 MPa
Limiting oxygen index 35 %
Elongation on break > 160 %
Ecotoxicological testings no toxic gases by combustion
Prototype compound: Polymer compound for cable, application app. 2 ECU/kg
cost: Polymer compound for adhesive application 0.6 ECU/kg

All industrial partners are able to use the project results- Grecian Magnesite as a producer of raw materials (MgO and $Mg(OH)_2$), Henkel, and NKT as end users. For NKT the area of application is primary cable insulation. Henkel already produces adhesives and sealants on a large scale. For Grecian Magnesite the production of $Mg(OH)_2$ flame retardant grade will be a basis for diversification to new, high added value products.

The project results belong to the" scientific fields B6 (Energy economics), EO 1 (Adhesive joining), E05 [Chemicals), E08 (Coating). E30 (Polymers)
They can be applied by the industrial sectors E04 (Construction industry). E06 (Electrical equipment), E 10 (Industrial chemicals).

Estimated market size for flame retardant polymers:

Year 1994	USA		EC		Japan	
	k tons	Mio Ecu	k tons	Mio ECU	k tons	Mio ECU
Thermoplastic Fire Resistant Polymers	4.076	4.897	3.864	4.637	1.743	2.090
Synth. Adhesives + Sealants	2.921	5.392	2.550	4.719	733	1.355
Total	6.997	10.289	6.414	9.356	2.476	3.445

Conclusions:

The cooperation partners have generated project results which are highly in accordance with the regulations of BRITE/EuRam. Prototypes of polymer compounds partially based on renewable raw materials have been developed as well as halogen free flame retardant compounds for the cable industry.
As described in the work program the process scale-up should be done up to 1 ton. During the project life time at NKT 7 tons of prototype compound were produced. Based on this encouraging result a scale-up step up to 100 tons should be possible in 1996. If regulations for banning PVC cable insulations take place in the near future approximately 10,000 tons of this polymer will be produced per year.
For adhesive applications formulations have been developed based on renewable raw materials - Nevertheless, the requirements for adhesives are high and the products and raw material are price sensitive- With a share strength of 5-6 Nm/m^2 and a price of 0.3 -0,6 ECU/kg the formulations developed are in the price range of existing compounds. A price and/or performance advantage has to be developed in the future. This part of the project could show that solvent free adhesives based on renewable raw materials can be formulated.
In the area of fillers for multiple applications a major advantage has been achieved. Magnesium hydroxides and -oxides are widely used in polymer applications. Within this project Magnesium hydroxides and -oxides have been developed based on natural magnesite and hydromagnesite which is also naturally occuring. Here a cost as well as a performance advantage is anticipated.

All partners started new research projects (mostly funded by the government) based on the promising project results. A lot of different routes for additional positive effects have been generated within this project. For the adhesive area dispersions were prepared with good performance and price features. Further investigations should show whether or not a competitive price/performance advantage can be reached. Based on the ILFPO project the partners are ahead of halogen free polymer compounds. Once a regulator-y decision by the government will be made, a substitution especial] y for cable insulation can be put into effect i n a short time.

Acknowledgement

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Abbreviations

Ring-open the epoxydized fatty acids methylester of soybean oil

Sample 1: by ethylenglycole in the molar ratio 1: 1

Sample 2: by ethylenglycole in the molar ratio 2:1

Sample 3: by Pluronic-PE 3100 (ethylenglycole terminated polypropylenglycol, BASF) in the molar ratio 2:1

Sample 4: by dimerdiol (dimeric fatty alcohol, C_{36}-dialcohol, Henkel) in the molar ratio 2:1 ,

Sample 5: by Poly-BD-R 45 HT (diacid of Polybutadien, Mw 2000) in the molar ratio 2:1

Sample 6: soybean oil/dimeric fatty acid ratio 1:0,8

Sample 7: " " " 1:1,0

Sample 8: " " " 1:1,3

Sample 9: linseed oil/ dimeric fatty acid ratio 1:2

Sample 10: " " " 1:1,5

Sample 11: Sojapolyol 85 (Henkel KGaA) / MSA

Sample 12: " 180 (Henkel KGaA) / MSA

Sample 13:" 85 (Henkel KGaA) / PSA

Sample 14:" 180 (Henkel KGaA) / PSA

Sample 15: castor oil / MSA molar ratio 1:2

Sample 16:" / MSA " " 1:1

Sample 17:" / PSA " " 1:2

Sample 18:" / PSA " " 1:1

Homolytic polyaddition of epoxydized fatty acid methyester

Sample 19: soybean oil basis, Mw about 20.000- 50.000

Sample 20: soybean oil basis, Mw about 100.000-.500.000

Comparable products by changing the acid groups (end groups)

Sample 21: dimeric fatty acids

Sample 22: trimeric fatty acids

Esterification of PPG (Mw 2000) by

Sample 23: PPG Mw 2000/ PSA 1:2

Sample 24: PPG Mw 2000/ MSA 1:2

Sample 25: PPG Mw 2000 / 13SA 1:2

Sample 26: PPG Mw 2000 / ADP 1:2

Sample 27: PPG Mw 2000 / dimeric fatty acid 1:2

ADP = adipic acid

BSA = succinic acid

EVA = ethylene vinyl acetate

FPI = fire performance index

LOI = limiting oxigen index

MFI = melt flow index

MSA = maleic anhybride

Mw = molecular weight

OHZ = number of OH-groups

PE = polyethylene

PPG = polypropylenglycole

PSA = phthalic anhydride

JZ = number of unsaturated groups

RHR = rate of heat release

SEA = smoke extinction area

SSA = specific surface area

Sz = number of acid-groups

THE = total heat evolve

TS = tensil strength

Vz = number of ester-groups

XRD = X-ray diffraction

MSA: maleic anhybride

PSA: phthalic anh ydride

BSA: succinic acid

ADP: adipic acid

Mw: molecular weight

PPG: polypropylengly cole