Executive Summary:

In the opening quarter of the project, the consortium decided upon three case-study parts to help drive the selection of suitable materials and mixing processes. For each part, the target fire-performance and mechanical properties were identified. Following this, key materials were identified and sourced for subsequent use by the partners in their development work.

At the beginning of Work Package 2, an important step was the characterisation of the Laviosa commercially available materials and subsequent comparison with the Southern Clay Products commercially available materials (which had previously been shown to improve fire performance in unsaturated polyesters, on a laboratory scale). This characterisation was completed early in Q2 and a suitable material from the Laviosa range was identified. After this, work focussed on the development, optimisation and production scale up of bespoke organomodified clays. These bespoke clays were produced on the kilogram scale in the laboratory, however further scale up on pilot plant equipment was not possible, due to issues of drying large-quantities of bespoke organomodified clay and subsequent registration of the product. Significant effort in the second Period of the project was therefore focussed on development of the 'one-pot' clay organomodification process; designed to overcome these issues. Once the 'one-pot' method had been optimised, further attempts were made to try and understand the factors which affect the fire performance of the final formulations.

Alongside this work, a series of trials were carried out in order to optimise, and subsequently scale up the mixing process. This section of work started with the screening of different mixing equipment, before moving on to optimise the rotor/stator process; firstly via the determination of optimum parameters for the 'standard' equipment and, subsequently, via the design, manufacture and testing of innovative rotor/stator elements. After successful demonstration on the laboratory scale, YTRON produced a large-scale prototype mixer. Large scale trials of this equipment were carried out at APC Composite, using a formulation developed by Gaiker, the 'one-pot' clay organomodification method developed by SHU and traditional fire retardants (TFR's) sourced by FEPS. The resultant material was then processed using techniques which had previously been investigated on a smaller scale by NetComposites with expert input from APC Composite. A second batch of material was later produced for the production of case study parts.

In Work Package 4, the delays in production of large quantities of bespoke organomodified clay meant that the formulation work was held up. However by the end of the project, two composite resin formulations and one coating formulation were produced, which were able to impart good fire performance - meeting many of the requirements for the rail industry in particular. One key factor which became apparent was that, in order to meet the strictest fire performance requirements, larger than expected quantities of TFR's were still required in the formulation. Furthermore, at these levels, the effect of organomodified clay is significantly reduced. Another factor was that the material is more difficult to process. Therefore Work Package 4 also involved significant effort towards optimising the production of test panels by hand layup, vacuum infusion and RTM. A more positive outcome was the discovery that the use of fire retardant coatings (developed by IRIS Vernici) allowed us to reduce TFR content in the composite resin formulation, thus aiding processability.

Finally, at the end of the project, three large-scale demonstrator parts were produced, and testing according to rail, construction and marine standards was carried out.

The health and environmental assessment work began with the preparation of a general process diagram, covering the lifecycle of the organomodified clay from production until end of life. Using this flow chart, a preliminary hazard analysis was carried out to identify the main areas of potential occupational exposure to nanoparticles and also to identify the main environmental aspects with potential significance. From here studies of the specific nanoparticle emissions during mixing, machining and spraying processes took place. Following on from these tests, a new risk management methodology was developed, which could be easily integrated by relevant companies as part of the overall risk management strategy. Alongside this, a review of the current state of the art with regard to toxic effluent emitted from fires was carried out. In summary, the acute toxicity of fire effluents is primarily determined by the regime of a fire (i.e. a fully ventilated fire with enough oxygen to boost the combustion process will lead to less smoke and lower amounts of acutely toxic species than an under ventilated fire with lack of oxygen and incomplete combustion). The influence of nanocomposites and nanoclays as particles on the regime of the fire has not been shown so far and is probably negligible.

In the area of dissemination, the project results were disseminated at several events across Europe and in China. This included poster presentations, invited talks, presentation of conference papers and the publication of a magazine article. At the end of the project, a workshop was organised to disseminate the final project outputs.

Project Context and Objectives:

Concept

The concept of Polyfire was to develop and scale-up techniques for processing halogen-free, fire-retardant nanocomposite materials and coatings based on unsaturated polyester resins and nanoclays. This would enable, for the first time, industrial scale production and exploitation of fire-retardant nano-filled polyester resins, and the replacement of hazardous halogenated fire-retardant additives. The materials would open up an extensive range of applications in a wide range of industrial sectors, not least construction and mass transport, where fire-retardancy and light-weight are critical. Our focus was on developing turn-key solutions that would be easily integrated into industrial companies, in particular SMEs in Western and Eastern Europe, thereby ensuring maximum impact and benefit to the European community.

Background

A total of 4,000 people are killed and 80,000 injured by fire every year in the European Union and the cost of fires to European countries is on average 1% of GDP. Although improvements have been made to the fire-retardancy of materials, there is still much room for improvement particularly regarding the toxic potential of the additives used to reduce both the spread of flame and the generation of smoke and off-gases. Conventional halogenated fire-retardant additives, such as chlorinated and brominated compounds, release gases on combustion which are toxic and corrosive, whilst high-load fillers such as alumina trihydrate (ATH) tend to reduce processability and impact strength. It is now well-established that nanofillers can significantly increase a wide range of polymer properties, including strength, stiffness and fire-retardancy. However, the vast majority of research has been conducted on thermoplastic polymers such as polypropylene (PP), polystyrene (PS) and polyamide (PA), and relatively little attention has been paid to thermosetting resins such as unsaturated polyester, despite worldwide consumption of over 1,600,000 tonnes . The ability of organomodified nanoclay to dramatically improve the fire-retardancy of polyester resins has been proved on a laboratory scale in a recent collaborative project, driven by 3 key Polyfire partners. Synergistic interactions between the polyester resin, nano-clay and other additives led to major mprovements in fire-retardancy, enabling UL94V0 and Euroclass D safety level to be achieved. The formulation was independently assessed and confirmed to be completely halogen-free. It was also discovered that, when using nanoclays, equal or better fire performance could be achieved with greatly reduced additive content and reduced cost. However, this halogen-free, nano-based material can currently only be made on a small scale, primarily due to the intricate nature of the nanoclay organomodification process and the need to ensure complete dispersion of the nanoclay during mixing. Significant breakthroughs are required to enable large-scale production and hence facilitate the exploitation by European industry. In addition, considerable further work is required to demonstrate the industrial application of the material and to fully assess the health and environmental impacts. A recent US market study states that US demand for nanocomposites will double to 411,000 tonnes by 2011 and that the technical issues concerning additive dispersion must be overcome to enable rapid growth. Given that the projected US demand for nanocomposites is also expected to increase 5 fold between 2011 and 2025, reaching 2 million tonnes, it is absolutely imperative that European industry is well-placed with a portfolio of competitive products in order to successfully penetrate this rapidly growing market. Consequently, the target of this project was therefore to develop and scale-up the nanoclay chemical modification process and the physical mixing and dispersion techniques which will deliver the production of the halogen-free, fire-retardant polyester resin on an industrial scale (typically 1000 tonnes per year). The up-scaled material would be used to produce fibre-reinforced composites and coatings, which will be thoroughly evaluated by a range demanding mechanical, physical and fire tests. Furthermore, the technology would be demonstrated by producing 3 industrial-scale case study components from key sectors such as construction, rail and marine. The partners would focus on developing materials which could be successfully applied in existing composite moulding processes and coating techniques to make integration easier and therefore maximise commercial uptake. Comprehensive health, environmental and economic impact assessments would be conducted, in parallel with the technical activities, to ensure that the materials and processes developed are sustainable.

Scientific & Technical Objectives

The overall goal of Polyfire was to scale-up the production of a novel, halogen-free, nanoclay-based, fire-retardant unsaturated polyester resin for use in nanocomposites and coatings. Within this broad goal, 5 primary objectives were set:

1. Optimise and scale-up the nanoclay organomodification process suitable for 10T/year
2. Develop and up-scale the nanoclay-resin mixing process suitable for 1000T/year
3. Optimise the nanocomposites and coatings to achieve target fire safety classifications including Euroclass C level in the EN-13501 European Normative for Construction
4. Prove the technical and commercial feasibility of the materials and processes by producing and testing 3 large-scale case study parts
5. Assess the health and environmental impacts of the nano-based material and processes

To achieve these primary objectives, the project was further sub-divided into a series of smaller tasks each with related technical objectives:

1. Select the nanoclay, organomodifications, synergistic resins, additives and mixing techniques that will enable large-scale production of the materials safely and economically
2. Optimise the bespoke organoclays for scale-up and to deliver the required fire performance and scale-up the production process suitable for 10T/year capacity
3. Develop at least 2 ways of introducing and dispersing the nanoclay into unsaturated polyester resin consistently
4. Investigate and develop a range of novel mixing techniques for dispersing the nanoclay consistently and economically on a large-scale, for example in-line high shear and sonication techniques
5. Develop a large-scale, prototype mixing machine suitable for 1000T/year capacity and verify the up-scaled mixing process

6. Develop at least 2 strategies for producing finished resins on a large scale, for example bespoke formulation using a synergistic base resin and adding a pre-dispersed masterbatch into a standard resins

7. Optimise the fire performance of nano-based fibre-reinforced composite parts to achieve target classifications in specific industry standard tests such as EN 13823 Single Burning Item for construction

8. Optimise the fire performance of nano-based coatings to achieve target classifications in specific industry standard tests including ISO 5658-2 Spread of Flame for construction and transport

9. Demonstrate the industrial feasibility of the nanocomposites and coatings developed by producing 3 large-scale case study parts from key industrial sectors and testing them to application specific requirementsIdentify and rank the health and environmental impacts associated with the nanoclay materials and processes and conduct in-depth studies on those with the highest risk potential

10. Develop a risk management methodology to minimise any potential negative impacts caused by the nanoclay particles and compare the health and environmental impacts of new and existing fire-retardant materials

11. Disseminate the findings and results of the project to target industrial users and the wider European community

Partners

NetComposites Ltd: Responsible for moulding nanocomposite test panels, developing the project website, building the industrial interest group and will assist in the mixing process development. They are also the project coordinator, responsible for technical and administrative coordination.

Laviosa Chimica Mineraria S.p.A: Responsible for the supply of standard and bespoke nanoclays to the project and will be responsible for scaling-up the modification processes.

Fundacion Gaiker: Participate in the development, production and testing of nanocomposites and coatings, using their extensive expertise in industrial polymer processing methods, material characterization and fire testing. Responsible for the formulation of nanocomposites resins and coatings, production of test panels by different manufacturing techniques and testing the fire properties and the mechanical properties in order to optimise the process.

YTRON Process Technology GmbH & Co. KG: Responsible for developing and trialling a range of novel mixing techniques for dispersing the nanoclay particles including in-line systems, innovative rotor/stator elements and ultrasound/sonication methods. They will optimise and upscale the most suitable technique and will build a prototype machine capable of 1000T/year output.

Scott Bader Ltd: Responsible for the development and manufacture of base polyester resins displaying synergistic interactions with nanoclays. They will be active in the development of styrene depleted and masterbatch systems and will assist with the scaled-up compounding of the final products and the health and environmental assessment. Exploitation of the project results will include using Scott Bader's established global presence in the fire retardant resins and gelcoat markets.

IRIS Vernici s.r.l.: Responsible for the formulation and optimisation of the nano-filled polyester coatings and will take a leading role in the industrial scale-up process. They will manufacture and test coatings case studies from the construction and rail sectors and will assist in dissemination through their links with the Procoat association.

APC Composite AB: APC are responsible for the development of large-scale case studies. They will focus on the development of resins for fibre-reinforced composite mouldings and will manufacture and test fire-retardant case study components for the marine and rail industries.

Dr Troitzsch Fire & Environmental Protection Service: Responsible for coordinating the activities regarding the selection of flame retardant systems, small and large-scale fire testing and optimisation of the fire performance. As work package leader, FEPS will coordinate and manage the dissemination activities including a project website, organising sessions at international conferences and workshops to inform the consortium members and the scientific community on the progresses of the project, and initiate publications in scientific and popular journals.

Sheffield Hallam University: Responsible for strategic direction regarding up-scaling of bespoke nanoclays and their dispersion into synergic resins. As the project progresses, they will provide knowledge-based support into the up-scaling of the mixing processes and the manufacture and characterisation of test panels and case-study parts, using their state of the art analytical facilities, which include XRD, FTIR, XRF, FEG-SEM, TEM, TG-MS and TG-GC-MS and modular rheometry.

Fundacion Tecnalia: In the Polyfire project, Tecnalia will focus on the health and environmental assessment of the nanoclay-polyester materials. They will conduct an in-depth review of the potential health and environmental impacts, coordinate specific studies on the nanoparticle emissions and develop a risk management methodology for the materials developed.

Procoat - Consorzio per la promozione dei prodotti vernicianti e ricoprenti: Responsible for providing advice on the formulation of nano-filled coatings and test methods and will assist with the demonstration phase, by drawing on the expertise of their industrial members. Their principal role will be in dissemination, including the management of the industrial interest group for coatings. With its network of partners and other companies regularly registering for its workshops and services and with its database of more than 1000 contacts in the coating business, Procoat is ideally poised for disseminating the project results whilst at the same time helping to keep and protect intellectual property rights.

Project Results:

The key results from each work package (and its' sub-tasks) is given below:

Task 1.1 - Selection of Fire-Critical Case Study Applications:

After discussion between the partners it was agreed that the three case-study parts would be as follows:

1. A balcony floor structure for the marine industry
2. A coated composite panel for the rail industry
3. A coated composite panel for the construction industry.

The balcony floor would need to meet reaction and resistance to fire requirements. It would also need to meet the necessary structural and aesthetic requirements. In order to do this, the part must be a sandwich structure, including a core material between two composite laminates. The rail industry part would be a wall or door panel and will be required to meet reaction to fire requirements. It will be a glass-fibre reinforced polyester part with a coating. The construction industry part would be a wall panel for a passenger lift, and would be required to meet reaction and resistance to fire requirements.

For each sector, an overview of the relevant limit values for heat release, flame propagation characteristics, smoke generation etc. during a fire scenario were collected, and would act as reference points during the subsequent material development work.

Task 1.2 - Identification of Materials

During this task, the partners defined the key materials which would form the basis of subsequent formulation development work. Broadly speaking, the key components were as follows:

1. Polyester resin
2. Commercially available bentonites and organomodified clays
3. Organomodifiers to be used to produce bespoke organomodified clays
4. Traditional non-halogenated flame retardants
5. Smoke suppressants
6. Char promoting agents

A shortlist of each material type was drawn up, and sample quantities ordered.

Task 1.3 - Identification of Mixing Techniques

In this task, the partners outlined the mixing techniques and parameters which would be investigated in the project. In the initial phases of the project, a range of mixing techniques would be studied including mechanical rotor/stator systems, mechanical three roll mills, ultrasonic/sonicating mixers, and combinations of different mixing methods. Once the best technique or techniques had been established, the project would focus on scaling up the technology and will develop a large prototype machine capable of producing 1000T/year.

Task 2.1 - Preparation, Optimisation and Characterisation of Bespoke Organomodified Clays

To achieve good fire performance in polyester-clay composites, correct dispersion of the clay in the polyester resin is critical. Mechanical mixing alone is not sufficient to overcome the combined effect of attractive forces between the clay platelets and incompatibility with the polyester resin.

By exchanging surface sodium ions on the clay with organocations (from a cationic surfactant) platelet - platelet separation and compatibility with the polyester resin are increased. Polymer molecules are then able to enter the clay gallery and begin to overcome the inter-layer attractive forces. Mechanical mixing is then used to provide the required level of dispersion.

Several organomodified clays are available commercially, and these have previously been shown to impart reasonable fire-retardant properties to polyester resins (when used in conjunction with other additives). However lab-scale studies have shown that, by using different cationic surfactants as organomodifiers, bespoke organomodified clays can be produced which impart significant improvements in fire performance, compared with the commercially available materials.

This task focused on up-scaling this lab-scale organomodification process and ultimately enabled the production of bespoke organomodified clays in kilogram quantities.

Task 2.2 - Scale-up, Optimisation and Safe Production of Bespoke Organoclays

The original focus of this Deliverable was the confirmation that Laviosa was able to produce bespoke organomodified bentonites in 100 kg quantities (which would contribute to producing tonne quantities of fire retardant resin).

Unfortunately, Laviosa reported difficulties in producing large quantities of this material because it proved much more difficult to dewater than they had previously experienced - that is the filtration rates that they achieved in the laboratory were very slow.

Despite attempting to work with several different bentonite grades and organomodifiers from several different suppliers (in different physical forms), the filtration rates in the laboratory remained too slow to risk attempting the production of larger quantities of the bespoke clay on the plant. The likely outcome was a severely blocked filtration system which would have caused unacceptable delays in the manufacture of their commercial organomodified clays.

Moreover, in addition to the dewatering challenges identified at Laviosa, there was an issue concerning the registration of the new organoclays and the safety concerns associated with shipping them between partners. The bespoke-modified clays do not have an EINICS number or an associated CAS number making it extremely difficult to complete the required documentation for shipping.

In order to address these issues a new line of investigation was initiated at SHU wherein the target was to produce bespoke-organomodified clay directly in the resin (rather than water, which is the solvent in the conventional ion exchange process). The bespoke-organomodified clay produced would NOT constitute a new product but would merely be an intermediate product in the overall production process. Consequently, the need for an EINICS registration, and hence the associated cost, would be avoided.

This process, termed the 'one-pot' process, although outlined as a possible area of investigation in the original Description of Work, took on a much larger dimension as a result of the inability to produce large-scale quantities of clay via the conventional ion-exchange route and further details of the process will be given in Task 2.4.

Task 2.3 - Optimisation of Diluent Content of Synergistic Resins

This task comprised production of the synergistic unsaturated polyester resin with varying amounts of monomer diluent and at plant-scale. The monomer diluent cross-links with the unsaturated polyester polymer on cure to create a matrix of high strength and stiffness for use in fibre composite applications, such as those under investigation in this work.

The aim of producing resins with differing diluent contents allows the possibility of pre-batching the fire retardant additives, such as the clay, into a slurry with the diluent, with the option of tailoring the amount of diluent based on the application and resin properties required. It should be noted that the unsaturated polyester resin, and most general unsaturated polyesters, are able to cure with a wide range of diluent contents and therefore typical commercial formulation development practice using these resins can involve optimization of the diluent content.

Task 2.4 - Dispersion of Nanoclay and Traditional Fire Retardants in Diluent and Introduction into Resins

The original focus of Task 2.4 was to optimize the pre-mixing of bespoke organomodified clays in monomer diluents in order to optimize the dispersion of the clay in the unsaturated polyester. However, since Laviosa were unable to produce the ion-exchanged bespoke-organomodified clays in the required quantities - due to dewatering issues -work in this task instead focused on production of the required formulations using the 'one pot' method. This method takes a natural Na-exchanged clay and combines it together with organomodifier directly in unsaturated polyester resins.

As mentioned in Task 2.2, evaluation of the one-pot method was an important aspect of the original work plan for Task 2.4, however its relative importance increased due to the challenges associated with dewatering the ion-exchanged bespoke-organomodified clay at Laviosa. In addition, the extent to which the resident inorganic cations (occupying the cation-exchange sites on the clay) are replaced by the bespoke-organomodifier was thoroughly explored in order to understand and eventually influence those factors which contribute to the successful production of bespoke-organomodified clays produced via the one-pot method, rather than by the conventional aqueous ion-exchange process.

Task 3.1 - Investigation of Innovative Mixing Systems

A major task in the Polyfire project is the incorporation of organomodified clays into a polymer resin, so as to improve the fire properties of the resin. To obtain optimum mechanical properties fully exfoliated nanocomposites are often desired. However, for best fire performance an intercalated structure is believed to be beneficial.

The most appropriate and robust technique to achieve the desired intercalated structure on an industrial scale is mechanical mixing, using high-shear- and rotor/stators-systems. Ultrasonic treatment, particularly with an ultrasonic frequency over 20kHz could also provide good dispersion of the organomodified clays. Ultrasonication generates alternating low-pressure and high-pressure waves in liquids, leading to the formation and violent collapse of small vacuum bubbles, a phenomenon called cavitation. This causes high speed impinging liquid jets and strong hydrodynamic shear-forces, which can help disperse particles such as organomodified clays.

An optimum mixing process was determined by a series of experiments. Both the influence of different types of machines and their variables - such as mixing head design, speed and flow - were studied. The first step was to study the equipment already present in the consortium. In the final large-scale mixing, a combination of these devices and / or the use of new equipment is envisaged.

In addition to the instrumentation itself, the efficiency of the mixing process depends largely on the composition of the mixture. Therefore, initial experiments focussed on simple formulations, so that the degree of clay intercalation/dispersion could be studied by XRD and, if necessary TEM.

The most effective and efficient technology was then taken forward for industrial-scale trials in order to realize large-scale material production - the emphasis being on maintaining the quality and consistency of the resulting dispersion, and the safe and economic production of fire-retarded resins.

Based on the laboratory-scale trials, it appeared that the most effective dispersion could be achieved using an in-line rotor-stator mixer. As this mixer works on a slurry-liquid concept, it will be necessary for a simple pre-dispersed to be carried out first. This pre-dispersion can be carried out using a lower-shear mixer.

Ultrasonication may assist in the dispersion of clay, when in conjuntion with the above techniques. However further studies would be required in order to obtain a definitive result.

Initial results with the triple-roll mill suggested that it was unlikely to provide the level of dispersion required. The technique also results in significant loss of volatiles from the polyester resin. However before this technique can be totally ruled out, further studies are requried.

Task 3.2 - Determination of Optimum Mixing Conditions

Several mixing techniques were previously evaluated (as part of Task 3.1) and the best performing ones selected for further study, with the aim of determining the optimum mixing parameters.

In addition to mixing, a section of work to ascertain the time dependency of the clay swelling process was carried out. The results indicated that there is little change in the viscosity profile during swelling and, on the whole, not enough evidence to demonstrate a measured difference in viscosity due to the clay swelling process. For this reason no definitive recommendation on the optimum swelling time can be given at this stage.

Further investigation of the triple roll mill technique proved that it will not be suitable for our application. Significant loss of volatiles when attempting to disperse clay in unsaturated polyester resin makes the technique unsuitable for a direct mixing process. Likewise the loss of the volatiles, coupled with their low viscosity, prevent their use in a pre-dispersion process.

The YTRON Y-Lab was studied in more detail; as both a one-step direct mixing technique and as part of a two-step slurry-liquid process. Initial investigation of the one-step technique during Task 3.1 proved inconclusive; therefore a further series of tests was carried out to look for variations in dispersion efficiency at the upper and lower performance limits of the Y-Lab. Results of these investigations revealed no discernible differences and, as such, it was concluded that use of the Y-Lab in a one-step technique was unlikely to provide the requisite level of clay dispersion.

Work in Task 3.1 suggested that the best mixing technique would be the two-step slurry-liquid mixing process; therefore the majority of work in Task 3.2 focused on improving and optimizing this technique. In particular, the incorporation of sonication, as an extra stage in the process, was extensively investigated. Two different sonication techniques were investigated and, despite appearing to have some influence on the mixing process (as evidenced by temperature rises and changes in the degree of sedimentation), neither was ultimately found to impart any improvement to the process.

As a result of these investigations it has been concluded that the best mixing method is a two-stage slurry-liquid process using the YTRON Y-Lab (at stage 1), followed the YTRON Z-Lab (at stage 2).

A further important outcome of Task 3.2 was the decision to use fire testing as the way of evaluating the various mixing processes, rather than X-ray diffraction.

Task 3.3 - Development of Innovative Rotor/Stator Elements for Large-Scale Mixing

The objective of this task was to design, manufacture and test innovative rotor/stator elements. The development of a novel adjustable gap rotor/stator device would enable the amount of shear to be varied thereby significantly improving the flexibility of the mixing system. This would enable the production of nano-filled resins with a range of additive contents and viscosities and the possibility of adjusting the organoclay dispersion during production to tailor performance and quality.

For the use of the new rotor/stator elements, the standard design YTRON Z-Lab had to be modified. To incorporate these modifications, a new Z-Lab was designed and manufactured. In typical operation, the Z-lab is an inline mixer, however due to the relatively small quantities of material being worked with at this stage of the project, an alternative operating method is being used.

The slurry or liquid is introduced via a funnel. After opening the valve below the hopper, the liquid or slurry flows through the hose into the open Z-machine and passes the rotor/stator set. Thereafter, the homogeneous product flows out of the outlet of the Z-machine. Important factors are the parameters of slot width, radial gap, rotational speed and flow rate. Additionally, the effect of multiple rotor/stator sets can be simulated by passing the product several times through the system.

The rotor element is connected via a shaft directly with the motor and can rotate at a maximum speed of 6960rpm. Relative to the rotor is the stator, which is screwed onto a thread in the housing. With this thread, the variable gap can be adjusted.

To examine the effect of this novel tooling, two studies were conducted, using formulations of differing viscosity. Both studies show that despite the use of different gap widths and radial gap, the fire test results remain very similar. However it was noted that in both studies the samples which were created with 1 mm slot width and 0.75 mm radial gap provided the best results in terms of combustion.

Task 3.4 Development of Prototype Large-Scale Mixing Systems

The large-scale mixing system developed consists of a YTRON Y and a modified YTRON Z machine with a pump located between them. The YTRON-Y machine is installed in a 200l tank, in which all components according to the recipe are mixed. While the liquid components are added directly into the tank, the solids are introduced via the funnel. After the YTRON-Y machine has mixed the components, the mixture will be transfer to the Z-machine with support of a pump. This machine will disperse the solids in the liquid. After the product has passed through the shearing zone, it is fed to the tank again. This recirculation alters flexibility as it allows repeated runs of the dispersing system to supply additional components and / or to achieve a better dispersing of the components. Inline pressure monitoring will be incorporated and investigated as a possible indicator of nanoclay dispersion. When the dispersing process is finished, the final product will immediately be transferred to 25kg drums.

Task 3.5 - Industrial Scale Mixing Trials

The industrial scale mixing equipment was used to produce approximately 100kg batches of fire retardant resin (according to two different formulations developed during the project). The viscosity of the resulting material was measured and compared with the same formulation, mixed using the lab-scale equipment.

In both cases it was found that the material had a higher low-shear-rate-viscosity after being passed through the YTRON Z - indicating better dispersion of the solid additives. Furthermore, neither sample showed increased viscosity at high shear rate, which would be indicative of styrene loss - suggesting that no significant styrene loss was occurring.

Overall it was found that there was very little difference in the viscosity of the two samples, and this reinforces our belief that the industrial-scale equipment produces the same results as the lab-scale equipment.

This material produced from these two batches was then used to produce the large scale case study components, plus some additional flat panels as required by several of the fire test standards.

Task 4.1 - Formulation of Nanocomposite Resins and Coatings

Formulations were designed and developed using: i) different traditional flame retardants and smoke suppressants, ii) different organomodified clays, iii) different glass fibre fabrics, iv) different polyester resins and process additives.

Each formulation was designed and manufactured in order to understand: i) the optimum percentage of clay to include, ii) the best type of clay to add, iii) the most effective ratio of different fire retardant additives, iv) the effect of traditional flame retardants.

The different processing methods (i.e. vacuum infusion, hand layup, RTM and coatings application) were also considered during the formulation development, by studying the effects of the various additives on viscosity, gel time and degree of cure.

Simple test panels were produced from each formulation (by vertical casting), and their fire performance (by cone calorimeter, smoke chamber, Bunsen burner for coatings and epirradiateur) and also their mechanical performance, using a universal mechanical machine.

Task 4.2 - Processing of Nanocomposite Parts

Small fibre-reinforced laminates were produced and the following observations were made:

1. Hand lay-up, RTM and infusion are processes that do not allow high viscosities. For this reason the total amount of powdered fillers to be added, cannot be very high
2. Formulations designed show medium/low reactivity at room temperature
3. Formulations developed show medium/high gel times in order to fill the corresponding mould during the manufacturing process
4. It is possible to produce nano-composite parts using a highly filled resin, by means of conventional equipment, when a low closely woven glass fabric is used: i) mat 450 g/m2 as a reinforcement in hand lay-up, ii) UNIFILO 450 g/m2 in RTM and infusion.
5. When the reinforcement to use is a denser one (i.e. 1700 g/m2 biaxial glass fabrics) the use of a flow-core layer between glass fabric layers is an interesting option.
6. Certain additives can affect gel times
7. Nano-composite parts manufactured need a suitable post curing time in order to obtain suitable mechanical properties.

For sandwich panels (featuring a balsa wood core) in particular:

1. The use of flexible balsa (rather than the rigid version) helps to wet-out the fibres, as the gaps between balsa blocks act as small resin channels
2. Multiple resin inlets, with a central vacuum take-off point, gave best wet-out of the fibres
3. It is possible to produce sandwich laminates using a highly filled resin, with the flowcore layer and gaps between balsa blocks both acting to distribute resin evenly across the laminate

Task 4.3 - Application of Nanocomposite Coatings

Using formulations developed in Task 4.1 several nano-composite coatings were manufactured and applied onto polyester laminate panels. Different panels were used as a substrate (with or without flame retardant treatment). Additionally coatings were applied on steel panels to allow a precise measurement of heat transfer.

For these preparations the following steps were carried out:

1. Formulation mixing: The different components (resin, additives, flame retardants, organoclays, etc) were mixed by means of a high speed mixer.
2. Basic parameters (viscosity, gel-time, density)were checked by, respectively, Brookfield, dry recorder and picnometer.
3. Coatings were applied on carbon steel panels, non-flame retarded polyester laminates and flame retarded polyester laminates (in both cases, the laminates were produced by hand layup using chopped strand glass mat)
4. For the polyester laminates a hole was drilled in the centre to accommodate a thermocouple during the cone calorimeter test in a way that the hot junction will be exactly located at the panel/coating interface.
5. All specimens were cast with a doctor blade in a controlled thickness cast, dried and hardened at room temperature then properly conditioned in a climatic chamber at 40°C, finally they were sent for fire testing (as part of Task 4.4).

Task 4.4 - Testing and Characterisation of Nanocomposites and Coatings

The composites and coatings developed through Tasks 4.1, 4.2 and 4.3 were tested and characterised in order to determine their fire performance and mechanical properties. A large amount of composite parts (made by hand lay-up, RTM and vacuum infusion), and coatings have been tested and only a brief summary of the general conclusions is provided here.

Concerning reaction to fire behaviour, cone calorimetry was used as a screening test because of the great amount of information that it generates (heat release, smoke production, CO/CO2 generation and mass loss kinetics) from a small test specimen (100 mm x 100 mm). Other fire tests were also carried out in order to obtain other fire characteristics required by the different standards (lateral flame spread determination, smoke chamber, FTIR, etc).

Mechanical characteristics were analysed by means of the Universal Test Equipment according to the suitable standards. Flexural and tensile characteristics have been checked and analysed.

The general conclusions can be summarized as follows:

1. It is possible to obtain intumescent coatings based on polyester resin and organomodified clays (plus the traditional blowing additives)
2. A novel formulation was produced, suitable for production of parts by hand layup, which can meet HL2/R1 requirement for rail sector (and EUROCLASS C-s2,d0 for building).

3. A second novel formulation, suitable for use in infusion and RTM processes, can fulfil HL1 for R1/R16 requirement in rail sector (EUROCLASS D-s2/s3,d0 in building)

4. However the addition our novel intumescent coating to the above formulation can significantly improve the fire performance, such that the final part can meet HL2/R1 or HL2/R7 requirements in the rail sector

5. In order to obtain suitable mechanical properties is necessary to guarantee: i) a good glass impregnation and ii) a suitable resin cross-linking process

Task 5.1 - Detailed Specification of Large-Scale Case Study Parts

At the start of this work package, it was agreed that the case study components would be as follows:

1. A rail interior panel (which would be a coated composite part

2. A construction industry interior wall cladding panel (which would also be a coated composite part)

3. A rail flooring panel (which would be a coated composite sandwich structure with a balsa wood core)

For each application case the boundary conditions vary and there are also various levels within each case that can or can´t be fulfilled. For instance the train industry has different demands if the vehicle is a tram, a short way train or a subway train, the material demands that are expected of the manufactured part are of course higher for a train that is harder to evacuate in case something were to happen.

Therefore for each of the case studies listed above, consideration was given to the fire properties, the weathering conditions the part would be subjected to and the structural demands. In addition, factors such as aesthetics and also material processability were considered.

Task 5.2 - Large-Scale Composite Component

A decorative interior wall cladding was produced and, as described above, it was decided that this part would be a coated composite component (rather than uncoated, as had originally been the plan for this deliverable).

The panel was produced by vacuum infusion, with the process initially being optimised using commercially available resin and non-fire retarded gelcoat, before then moving onto using a resin formulation developed during the project plus a standard gelcoat, before finally producing the demonstrator part using the novel resin formulation plus an intumescent coating, developed in Work Package 4.

Task 5.3 - Large-Scale Coated Structure

A rail interior wall cladding panel was produced and, as described above, this part was also to be a coated composite structure (rather than a non-composite substrate, as had originally been planned).

The first stage in the production of this part was to design a mould, from which a composite tool could be made by a hand layup process. The composite tool would then be used to produce the final part. This is a common approach for the production of composite parts.

As with the wall cladding panel, this rail interior part was produced by vacuum infusion, with process optimisation first being carried out using commercially available resin and coating, before producing the final demonstrator part using a novel resin formulation and novel intumescent coating, both of which developed in Work Package 4.

Task 5.4 - Large-Scale Composite Structure with Coating

A rail interior flooring panel was produced and, as described above, this was a sandwich structure featuring a balsa wood core.

The manufacturing of the train floor was made on a steel vacuum infusion table that is suitable for making flat panels. The process was hand lay up for the coating and then a vacuum infusion for the laminate.

The processability of the novel resin was found to be very good and also uniform over the different production runs.

Test results showed that in general fire resistance properties of the material are good (this will be discussed in more detail in Task 5.5). Other properties of the material that has to be fulfilled are the aesthetic properties and the weather resistance of the material. The general reaction to the coated parts has been overall positive indicating that the material is useable for visible panels in all kinds of constructions. The test results are of course a subjective assessment, but the coated materials perform and look much like an ordinary gel coat that is the material that we have to relate to. Our opinion is therefore that this coated material will work as well as any coating that is out there on the market.

The fibre content of the test specimens indicates that this material is very good for construction purposes, making it useable in all kinds of parts of the construction; this was not a property that we expected. The general problem with other flame retarded polyesters are that they are filled to such an extent that the processability of the material is impaired, and that it therefore is difficult to manufacture a high tech laminate from those polyesters. Our assessment of this resin is therefore very good.

Task 5.5 - Testing and Analysis of Large-Scale Prototypes

Two different laminates were produced to allow both reaction to fire and resistance to fire testing. The laminates were as follows: A 5.5mm thick gel-coated laminate (4mm laminate + 1.5mm gel-coat) and a sandwich panel, comprising two 5.5mm laminates (as above, however the coating was only applied to one side) either side of a 25mm balsa wood core.

In the reaction to fire tests the 5.5mm thick gel-coated laminate showed:

1. Very low values related to heat release: i) MARHE (56,76 kW/m2 and 26,35 kW/m2 depending on the radiation supported 50 kW/m2 and 25 kW/m2), ii) FIGRA 0,2 and 0,4 MJ (64,5 W/s), and iii) total heat release THR (6,3 MJ). Taking into account these results the product could be considered non-flammable (this sort of products does not generate a fire)

2. Low flame propagation in the SBI (no lateral flame spread LSF), vertical radiant panel (CFE), flooring (horizontal) radiant panel (CHF) and small burner test. This fact means that the product designed and developed will not involve other adjacent products in a possible fire.

3. No debris or drops fall from the specimen in the vertical tests such as SBI and vertical radiant panel. That fact means that the adhesion between the coating and the composite part has been a suitable one and the product will not involve a floor in a possible fire.

4. Medium amount of smoke generation. This fact is the most negative one in the developed composite part. Nevertheless, it can be improved using smoke suppressants such as zinc hydroxystannate or improving the intumescent layer.

Therefore concerning the suitability in the different sectors, the product designed and developed:

1. Can be used as a floor covering in all kinds of buildings including Hospitals.

2. Almost fulfils all of the requirements for a wall and ceiling covering in all kinds of buildings (except Hospitals). The only value to improve lightly is the Total Smoke Production (TSP 600s) = 205.4 m2, being the requirement TSP 600s < 200 m2.

3. Can be used for interior applications in railway sector: i) as wall and ceiling covering in tramps (HL1) and long way trains (HL2), and ii) as a floor covering in tramps (HL1).

4. Can be used for exterior applications in railway sector: i) as external body shell in tramps (HL1), ii) as an external body roof in tramps (HL1) and long way trains (HL2), and iii) as an external cab housing in tramps (HL1).

5. Cannot be used in naval sector neither in bulkheads, wall and ceiling coverings nor in floor on primary deck coverings.

For the resistance to fire test, the results were very encouraging and suggest that a sandwich laminate with 3mm coating could achieve an REI 30 rating in a real classification test.

Task 6.1 - Identification of Health and Environmental Impacts

To date there is no clear evidence for the presence of nanoparticles in the unsaturated polyester composites produced in this project. The x-ray diffraction evidence suggests that an intercalated nanocomposite or a microcomposite is being produced. Nevertheless, the position being taken is that there is the potential to generate nanoparticles during organoclay production, the mixing of organoclay with the resin and when the moulded component is cut or sanded, for example. Therefore, this health and environmental assessment is designed to promote safe industrial production systems. Future work within this work package will seek to evaluate the type and nature of any nanoparticles generated, identify adequate control measures and educate relevant individuals and companies of adequate control measures.

The analysis of the potential H&E impacts has been performed with two tools, a Preliminary Hazard Analysis (PHA) and an Impact Matrix (IM). The results of this study shows that production is the life cycle stage where the potential hazard to particles exposure may be higher, linked to the manipulation of powder nanomaterials.

Task 6.2 - Specific Studies of Nanoparticle Emissions

This deliverable collected the results achieved in the experimentation performed to characterize the release of particles in four selected exposure scenarios (ES).

In summary the results of the studies performed suggested that there is no additional release of nanomaterials (considering number concentration and size distribution) different from the conventional operations without organoclays except for the mixing process. Data suggested that the process of handling an organomodified clay (ES2) or a non-organomodified clay can produce the release of particles bigger than 100 nm, which would be out of the nanomaterial classification (based on the EC recommendations for NM definition); additionally, data showed that the release can be higher for the non-modified clay (i.e. sodium clay) than for the organomodified clay. Results related to the process of mixing an organomodified clay with a viscous liquid like the resin (ES1) can produce the release of very few particles. Results further suggest that the viscosity in the mixing process can be a relevant factor for the release of particles. The process of machining (sawing) a nanocomposite filled with organoclays (ES3) and a vertical cast without filler can produce the release of small particles similarly in both materials. Two processes were tested, sawing and drilling.

Comparing both, the drilling process produced the release of fewer particles than the sawing one, suggesting that different machining process and conditions can produce different exposures difficult to predict. Finally the process of spraying a coating (resin) filled with organoclay (E4) and a coating without filler can produce the release of small particles, most of them smaller than 100 nm in both cases. Data achieved in the conditions of our tests suggested that the coating filled with organoclays can produce slightly less particle concentration and bigger particles, suggesting that the increase on the viscosity may produce a decrease in the number concentration and size of the particles released. However, different spraying equipment and conditions can produce a different release behaviour that would need additional research.

To complement this experimental studies on nanomaterial release, a review of the literature on the toxicity of organoclay particles and on its potential impact on fire effluents was performed. Scarce literature on toxicity of organically particles showed no conclusive results; however, recent studies performed in the Nanopolytox project may suggest that moderate toxicity can be due to the organomodifier. Similarly, preliminary conclusions from the few publications available are that the acute toxicity of fire effluents is not increased by nanocomposites.

It is important to make here a consideration about the risk. As it is known, risk is the product of two factors: exposure and hazards. In this experimental work we have measured the release of NMs that could be useful to extrapolate workers exposure. However, as the literature showed, there are no conclusive data regarding the toxicity of the organoclays yet. As a derivation, considerations about the risk of the new processes involving organoclays could not be done at the present time.

Task 6.3 - Risk Management Methodology

This task provided a method for companies producing new fire retardant composites and/or coatings containing organomodified clays to implement a system for the management of potential risks arising from the use of organomodified clays. This method is framed in the standard OHSAS 18001 Occupational health and safety management systems-Requirements.

In previous tasks, two main activities were performed. Firstly, potential exposure scenarios to organomodified clays were identified through the analysis of industrial processes (included in deliverable D6.1). Secondly, experimentation was performed to analyse the release of nanomaterials in selected scenarios (included in D6.2). With these inputs, this document proposes a system to manage the potential occupational risks arising from the use of organomodified clays in the new industrial process.

It is important to point out that bentonite clay (a naturally occurring material) is not expected to pose a risk to health (except for the possible content on crystalline quartz). However there is currently inconclusive evidence on the toxicity of manufactured nanomaterials (in general) and also on that of organomodified clays. The position taken in this document is to consider organomodified clay as a manufactured nanomaterial (MNM) and provide measures to manage and control the potential risks collected from the actual state of the art.

Task 6.4 - Comparison of Health and Environmental Impact of New and Existing Materials

In this task a list of indicators were proposed to compare the technologies used to produce traditional halogenated fire retardants with those used to produce the new organomodified clay-based products.

The comparison of the indicators proposed in this document suggests that the substitution of halogenated compounds by organomodified clays to produce FR leads to materials which have a lower impact on health and the environment. Indicators showed a decrease on potential impacts of new materials, although we must recognize that there remain uncertainties regarding the potential effects of nano-sized organomodified clays.

Moreover, indicators proposed in this document may be customized and used by industry to monitor the performance regarding environmental and occupational issues, through their integration in their risk management system.

Task 7.1 - Project Website

A domain name (www.polyfireproject.eu) and skin for the project website were purchased at the start of the project and it was agreed by the Partners at the POLYFIRE kick off meeting that the website should have two separate sections: Public and Private. The public section would contain the following pages: Homepage, Background, Polyfire Project, Partners, News, Contact and Links. The private section would be used for project management purposes.

Initial content was subsequently added to the Homepage, Background, Partners, News and Contacts sections of the website. This content was approved by the Partners, before being made available for public viewing. A brief summary of the content available on each page is given below:

Homepage:

This section gives a short general overview of the aims of the project. It also includes the logos of the European Commission and the Seventh Framework Programme.

Background:

This page is split into three sections; Unsaturated Polyesters, Fire-retardant Coatings and Nanocomposites and Fire Retardancy. Each section gives a more detailed description of the role of each component.

Polyfire Project:

This page gives a more specific outline of the aims and objectives of the project.

Partners:

Includes a short description of each of the Partners involved in the project. It includes contact details and links to partner websites.

News:

This page will be regularly updated with news relevant to the project This is likely to include conferences, workshops, publications and industrial activities.

Contact Us:

Contact details for NetComposites (as project coordinator) are listed in this section. Alternatively, there is a contact form which can be filled in online and submitted to NetComposites.

Links:

As the project progresses, this page will become populated with links to websites of interest to the project.

Task 7.2 - Industrial Interest Group and Exploitation Plans

The Industrial Interest Group IIG has been built up to around 90 interested parties during the Polyfire project. The objective was first to present the project, then to discuss the main non-confidential project outputs, and to exchange ideas regarding scientific and technological information for creating a basis for their exploitation and potential commercialization. The final Polyfire workshop on 31 August 2012 was very successful and has fuelled the interest of the participants, particularly for intumescent gelcoats.

In the Polyfire project, some very interesting results were achieved, regarding the production of organomodified clays ('one pot method'), a new dispersion technology for the organomodified clay, and the development of FR formulations and intumescent gelcoats for coatings with organomodified clays for unsaturated polyester resins used in rail vehicles and building construction. However, the consortium is of the opinion that the IP generated is either not yet patentable, or would be better protected by confidentiality agreements. It was decided by the respective consortium members having developed these new technologies that no activities would be started to apply for patents at this point in time; however, this may be the case later on.

Task 7.3 - Dissemination Activities

The dissemination activities have been promoted in the Polyfire website, in various publications, conferences, workshops and fairs. After completion of the website in M3, it has been regularly refined and updated until the end of the project. It has been visited and viewed over average compared to other science sites of similar size.

A series of publications, a web clip and an interview on the Polyfire project and its results were made. The Polyfire project and the results obtained were successfully presented with papers and posters at more than 25 conferences, workshops and fairs in 7 European (BE, CZ, DE, ES, FR, IT, UK) countries, Turkey and China from September 2009 to August 2012. These dissemination activities boosted the interest of many professionals in the field to join the Industry Interest Group IIG.

Potential Impact:

Impacts:

At the start of the project, it was anticipated that our main output would be the knowledge, materials, process techniques and prototype equipment to produce large quantities of nano-filled, fire retardant polyester material cost effectively and with consistent quality. In addition, we anticipated that the project would generate a range of fire-retardant fibre-reinforced composites and coatings tested to international standards and specifications, three demonstrator components allied to key industrial sectors (e.g. construction, rail and marine) and health and environmental assessments and documentation.

The results summarised in the earlier section of this report show how we have gone a long way to achieving these goals and, in the case of rail and construction industry applications in particular, we have developed materials that could offer real commercial benefits.

As such, we can be satisfied that the main impacts are indeed as stated at the beginning of this project, i.e. the availability of a halogen-free, fire-retardant polyester resin on an industrial scale for European industry within 2-5 years of the end of the project, with the potential to save thousands lives in fires and significantly reduce environmental impact.

Dissemination Activities:

The Industrial Interest Group (IIG) was created to inform a wide range of professionals in industry and academia of the Polyfire project and to discuss the main non-confidential project outputs of the project through posters and papers presented at conferences and dissemination workshops. Many professionals, who attended the Polyfire dissemination events, joined the IIG. The number of IIG members finally grew to around 90 individuals. The IIG members were kept informed by mailings of the presentations describing the project progress and encouraged to exchange ideas regarding scientific and technological information necessary for the successful completion of the project.

The major events, where professionals involved in flame retardancy, polymers, composites and coatings showed interest and later joined the IIG, were:

1. Nanocentral Nanomaterials for Low Carbon Transport event, 2 March 2010, Loughborough, UK

2. LNE Technical Workshop 'Nanomatériaux: de l'industrie au Consommateur', 4 March 2010, Paris, France

3. European Initiative 'Nanofutures. European Technology Integration and Innovation Platform in Nanotechnology', 15-16 June 2010, Gijon, Spain

4. 2010 International Symposium on Flame-Retardant Materials & Technologies ISFRMT2010, 17-20 Sep 2010, Chengdu, China

5. Composites Engineering Show, 29 Sep 2010, Birmingham, UK

6. K2010 International Plastics Fair 'Kunststoffe', 1 Nov 2010, Düsseldorf, Germany

7. NANOSAFE 2010 Conference, 16-18 Nov 2010, Grenoble, France
8. SKZ Conference on Trends in Fire Safety and Innovative Flame Retardants for Plastics, 24-25 Nov 2010, Würzburg, Germany
9. 2nd International Conference on Flame Retardants, 15-16 May 15-16 2011, Guangzhou, China, and following Chinaplas Fair, 17-18 May, 2011, Guangzhou, China
10. 13th European Meeting on Fire retardant Polymers, FRPM11, 27-30 June 2011, Alessandria, Italy
11. Composites in Fire Conference, 9 June 2011, Newcastle University, UK
12. RECTA 2011 Science and Technology of Aerosols, 27-29 June 2011, Madrid, Spain
13. EUROCLAY 2011, 26 June -1 July 2011, Antalya, Turkey
14. Composites Engineering Show 2011, Nanomaterials in Composites, NEC, 9-10 Nov 2011, Birmingham, UK
15. Fire Engineering for New-Build FRP Structures 2011, BRE, 22 Nov 2011, Watford, UK
16. JEC Europe, Composite Show Conferences, 27-29 March 2012, Paris, France
17. Polyfire Industrial Interest Group Workshop at the Shanghai FR Conference, 17 April 2012, Shanghai, China
18. Nanostructured Polymers and Nanocomposites Conference, 24-27 April 2012, Prague, Czech Republic
19. 10th International Conference on Occupational Risk Prevention, 23-25 May 2012, Bilbao, Spain
20. NanoFormulation2012, 28-31 May 2012, Barcelona, Spain
21. Polyfire Industrial Interest Group Workshop at the UK Research Office, 31 August 2012, Brussels, Belgium

FEPS has regularly updated the list of the IIG parties. A letter sent to the former IIG members of the Surefire project regarding their involvement in Polyfire IIG showed positive response. Since the start of the Polyfire project, the IIG has grown to currently 87 interested parties from 17 countries (Belgium, Czech Republic, China, Ethiopia, France, Germany, Italy, Japan, Korea, Malaysia, Netherlands, Poland, Russia, Spain, Sweden, Switzerland, United Kingdom). The members are working in the following fields:

1. Cable producers
2. Coatings
3. Composites aerospace
4. Composites rail vehicles
5. Composites ship building

6. Construction industry
7. Defence industry
8. Electrical engineering & electronics
9. Electricity distribution
10. Fabrics coating
11. Fibres pultrusion
12. Fire technology research
13. Fire testing
14. Flame retardants producers
15. Flame retardants research
16. Flame retarded natural fibres
17. Glass fibre producers
18. Nanocomposites in medicine
19. Nanoparticles research
20. Nanoparticles toxicology
21. Polymer compounders and masterbatchers
22. Polymer producers
23. Pultrusion
24. Upholstered furniture

From different events, where the Polyfire project has been presented, several parties, particularly from China, were quite interested in the IIG activities and joined. For this reason, FEPS organized a Polyfire IIG Workshop in Shanghai on 17 April 2012 in conjunction with the 3rd International Conference on Flame Retardants, and the Chinaplas 2012. Around 30 participants joined the Polyfire IIG workshop, where the Polyfire results achieved and dissemination activities were presented by Jürgen Troitzsch, FEPS. A discussion on scientific, technological and potential commercial opportunities from the Polyfire project took place. One example was the emerging interest in flame retarded, halogenfree thermosets instead of thermoplastics in injection moulding, and the role of halogenfree formulations containing organomodified clays. The benefits of injection moulded thermosets are seen in improved corrosion resistance, better flame retardancy, good dimensional stability, high temperature resistance, and lower cost compared to thermoplastics.

In light of the success of the China Polyfire IIG workshop, a second and final Polyfire workshop has been organised. The latest achievments of the Polyfire project were presented in the final Polyfire workshop, which took place after our last Q12 meeting in Brussels, the last day of our project, on 31st August 2012. The around 20 participants were very interested in our project results, which may lead to future contacts for PUDF. The main results from our work were shown and presented by Ben Hargreaves, Netcomposites, Chris Breen and Francis Clegg, SHU, Achim Dehnz and Johannes Trauwitz, Ytron, Claudio Pagella, Iris Vernici, and Jürgen Troitzsch, FEPS. A discussion on scientific, technological and potential commercial opportunities from the Polyfire project took place. The agenda of the programme was:

1. Polyfire Project Introduction
2. Background
3. Outputs of previous research
4. Polyfire project objectives
5. Consortium
6. New ways of dispersing organomodified clays
7. Intumescent gelcoats containing organomodified clays
8. Case study for railways and fire tests
9. Health and Environment assessment from nanoparticle emissions
10. Conclusions and outlook
11. Final discussion on scientific, technological and potential commercial opportunities

Intumescent gelcoats containing organomodified clays was the topic which attracted most attention from the producers of flame retarded unsaturated polyester components. The good fire safety results obtained in the case studies for railways and building also led to lively discussions and great interest.

Exploitation Strategy Seminar (ESS):

An EU sponsored Exploitation Strategy Seminar (ESS) was requested for POLYFIRE and confirmed by the Commission. It took place in Alessandria, Italy, on Nov 30th, 2010. The seminar was managed by Dr. Caocci, Cimatec. A documentary search document previously produced for Polyfire had been distributed to the Polyfire consortium members and discussed at the Q4 meeting in Bad Endorf (Ytron) in September 2010. The synthesis report for exploitable results generated by Dr. Caocci was then distributed and filled in by the Polyfire consortium members, and finalized by FEPS for the ES seminar, which turned out to be quite successful. The final version of the ESS report was sent by Dr. Caocci to the Commission in December 2010.

In the seminar, background, foreground information and exploitation claims have been characterised, the risks at exploitable result level were defined and an action plan to manage these risks developed. In the synthesis of the report, the key points of the project were summarised, the project assessed and recommendations given by Dr. Caocci. Some potential problems regarding co-ownership of results, joint exploitation strategy and technical risks were identified and recommendations given to minimize any impact on the results exploitation. A plan for using and disseminating the foreground (PUDF) to be completed and kept update by the consortium was developed. The ES Seminar findings developed are taken as the basis to better manage the projects results exploitation in the course of the Polyfire project.

Exploitatable Results

In the Polyfire project, some very interesting results were achieved, regarding the production of organomodified clays ('one pot method'), a new dispersion technology for the organomodified clay, and the development of FR formulations and intumescent gelcoats for coatings with organomodified clays for unsaturated polyester resins used in rail vehicles and building construction. However, the consortium is of the opinion that the IP generated is either not yet patentable, or would be better protected by confidentiality agreements. It was decided by the respective consortium members having developed these new technologies that no activities would be started to apply for patents at this point in time; however, this may be the case later on. In the meantime, IRIS Vernici and Scott Bader will continue discussion as to how best to exploit the newly developed coating technology, whist SHU will look for further opportunities (both commercial and funded) to demonstrate the 'one-pot' method in other resin systems.

The exploitable results list based on the outcome of the ES seminar has been refined during the course of the Polyfire project and the key results are summarised below.These results in principle might need after the project further R&D, prototyping, engineering, validation etc. before they become commercially exploitable.

1. One-pot clay organomodification process (SHU)
2. Mixing and dispersing equipment (product) (YTRON)
3. Mixing and dispersing process and parameters (process) (Gaiker)
4. Knowledge of fire retardant additives, formulations (FEPS)
5. Fully formulated polyester-clay resins (Scott Bader)
6. Flowcoat & gelcoat: Polyester-clay additives intumescent coatings (IRIS)
7. Knowledge of how to use the resins in composite processes (NetComposites)
8. Prototype demonstrator parts and associated performance test data (APC)
9. Training material/knowledge (Procoat)
10. Health and environmental risk management methodology and documentation (Tecnalia)

List of Websites:

http://www.polyfireproject.eu