

# **Environmentally friendly flame retardant used in PP/ cellulose composite material**

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<b>EXAMENSARBETE</b>	
Arcada	
Utbildningsprogram:	Plastteknik 2007/ Plastics technology 2007
Identifikationsnummer:	9302
Författare:	Christian Östman
Arbetets namn:	Användning av ett miljövänligt flamskydd i ett PP/cellulosa baserat kompositmaterial.
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Uppdragsgivare:	Deflamo Ab
<p><b>Sammandrag:</b>  Deflamo Ab är ett svenskt företag som tillverkar och marknadsför ett miljövänligt och biologiskt nedbrytbart flamskydd under namnet Apyrum till den europeiska marknaden. Syftet med projektet var att kombinera flamskyddet med UPM:s miljövänliga kompositmaterial (ForMi GP) som baserar sig på cellulosa och polypropen. Försök gjordes för att ta reda på ifall Apyrum fungerar i kompositen och vilka mängder det krävs för att uppnå önskade flamskyddsegenskaper. Också andra flamskydd användes i projektet. De två materialen blandades tillsammans m.h.a. en extruder och för att få lämpliga provexemplar för vidare test användes en formsprutningsmaskin. Brandtest gjordes hos Deflamo i Sverige och hållfasthetstest gjordes på Arcada i Finland. Resultaten var inte önskade på grund av att flamskyddet klimpade sig i extruderingskedet vilket ledde till att flamskyddsegenskaperna inte kom fram på korrekt sätt i brandtesterna. Problemen med att få en jämn och fin spridning av Apyrum i kompositen borde lösas för att få en klar bild av flamskyddsegenskaperna. Detta kunde dock inte lösas under projektets gång.</p>	
Nyckelord:	Deflamo, Apyrum, flamskydd, miljövänlig, UPM, ForMi GP
Sidantal:	46
Språk:	Engelska
Datum för godkännande:	18.12.2012

DEGREE THESIS	
Arcada	
Degree Programme:	Plastteknik 2007/ Plastics technology 2007
Identification number:	9302
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Title:	Environmentally friendly flame retardant in a PP/ cellulose based composite material.
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<p>Abstract:</p> <p>Deflamo Ab is a Swedish company that manufacture and markets the environmentally friendly and biodegradable flame retardant Apyrum to the European market. The aim with this project was to blend the flame retardant with UPMs environmentally friendly composite material (ForMi GP) that is based on cellulose and polypropylene. The goal was to find out the compatibility between the materials and which quantities of Apyrum that is needed to achieve wanted flame retarding properties. Other flame retardants were also used during this project. First the materials were compounded together with an extruder and then a injection molding machine was used to make specimens suitable for further tests. Fire tests were made at Deflamo in Sweden and the tensile tests were made at Arcada in Finland. The obtained results were not successful as the flame retardant formed lumps during the extrusion step that resulted negatively on the flame retardance properties during the fire tests. This problem should be solved in order to find out the true flame retardance properties and function of Apyrum in the composite. This could nevertheless not be solved during the project.</p>	
Keywords:	Deflamo, Apyrum, flame retardant, environmentally friendly, UPM, ForMi GP
Number of pages:	46
Language:	English
Date of acceptance:	18.12.2012

OPINNÄYTE	
Arcada	
Koulutusohjelma:	Plastteknik 2007/ Plastics technology 2007
Tunnistenumero:	9302
Tekijä:	Christian Östman
Työn nimi:	Ympäristöystävällisen palonsuoja- aineen käyttö PP/ selluloosa komposiittimateriaalissa.
Työn ohjaaja (Arcada):	Mirja Andersson
Toimeksiantaja:	Deflamo Ab
<p><b>Tiivistelmä:</b>  Deflamo Ab on ruotsalainen yritys joka valmistaa ja markkinoi ympäristöystävällistä ja biohajoavaa palonsuoja- ainetta Apyrumia euroopassa. Tämän projektin tarkoitus oli sekoittaa Apyrumia UPM:n ympäristöystävällisen komposiittimateriaaliin (ForMi GP) joka koostuu selluloosasta ja polypropeenista. Tavoitteena oli selvittää materiaalien yhteensopivuus sekä tarvittavat määrät Apyrumia palonsuojausta varten. Myös muita palonsuoja-aineita käytettiin projektin aikana. Materiaalien sekoittamiseen käytettiin ekstruderia ja lopullisten testikappaleiden tekemiseen käytettiin ruiskuvalukonetta. Palokokeet tehtiin Deflamon tiloissa Ruotsissa ja venytystestit tehtiin Arcadassa Suomessa. Haluttuja tuloksia ei saatu sillä ekstruusiovaiheessa palonsuoja-aine muodosti klimppejä mikä vaikutti kielteisesti palo-ominaisuuksiin palotesteissä. Tämä ongelma kuuluisi ratkaista jotta saataisiin selville Apyrumin todelliset vaikutukset ja palonsuojaomaínaisuudet UPM:n komposiitissa. Tätä ei kuitenkaan pystytty ratkaisemaan projektin aikana.</p>	
Avainsanat:	Deflamo, Apyrum, palonsuoja aine, ympäristöystävällinen, UPM, ForMi GP
Sivumäärä:	46
Kieli:	Englanti
Hyväksymispäivämäärä:	18.12.2012

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## ABBREVIATIONS

FR	Flame retardant
BFR	Brominated flame retardant
PBDE	Polybrominated diphenyl ether
PBB	Polybrominated biphenyl
PCB	Polychlorinated biphenyl
decaBDE	Decabromodiphenyl ether
TBBPA	Tetrabromobisphenol A
HBCD	Hexabromocyclododecane
POP	persistent organic pollutant
PP	Polypropylene
PVC	Polyvinylchloride
GP30	UPM ForMi GP30 granule
GP40	UPM ForMi GP40 granule
GP50	UPM ForMi GP50 granule
ATH	Aluminum hydroxide, Al(OH) <sub>3</sub>
RPM	revolutions per minute
UPM	United Paper Mills
RoHS	EU directive, Restriction Of Hazardous Substances
WEEE	EU directive , Waste Electrical and Electronic Equipment
REACH	Registration, Evaluation, Authorization and Restriction of Chemicals
EFRA	European flame retardant association

# 1. INTRODUCTION

Many plastic materials have a tendency to start burning if they are exposed to fire. Fires kills and injures people and destroys material possessions. In 2005 fires killed 85 people in Finland and the direct economical losses were 225 million € (Geneva association 2008). To prevent a fire from starting and continuing so called flame retardants are often used in e.g. polymers and textiles. The production and usage of different polymers and composites is growing steadily which also means a larger need for flame retardants. Deflamo Ab is a Swedish chemical company that manufacture and market the environmentally friendly flame retardant Apyrum. UPM has developed a new cellulose and polypropylene based composite material suitable for injection molding. Thus this more environmentally friendly composite decreases the need and use of oil based raw materials. A certain percentage is replaced with the renewable raw material cellulose. Products from the two earlier mentioned companies were used in this project.

One main group of flame retardants used are so called halogenated flame retardants. These are organic compounds containing bromine or chlorine atoms. Some brominated flame retardants (BFRs) have been banned in Europe due to their toxicity and capability to bioaccumulate in living organisms, including humans. Some BFRs show characteristics of POPs (persistent organic pollutants)(Janssen 2005 p. 26). The European directive RoHS has banned the use of some brominated substances in electronics in Europe and the WEEE directive instructs the removal of brominated FRs in electrical and electronic waste recycling. Chemicals need to be registered in REACH (registration, evaluation, authorization and restriction of chemicals) where they are first evaluated and later decisions on approval are made. REACH is established to protect the safety of humans and the environment against the risk that chemicals might bring (Brominated Science and Environmental Forum, BSEF).

Brominated flame retardants have been found e.g. in our food, in us humans and in breast milk that babies drink. In animal studies it has been showed that some BFR effects are neurobehavioral toxicity, thyroid hormone disruption and possibly cancer. Other evidence suggest that BFRs might also contribute developmental effects, endocrine disruption, immunotoxicity and long term effects (Janssen 2005 p. 19). In a study made by researchers at the University of California at Berkley it was shown that

prenatal and childhood exposure to PBDEs lead to decreased fine motor coordination and cognitive development in early school age children (Eskenazi *et. al.* 2012). New regulations, changing attitudes and growing environmentally friendly values have contributed in an increased interest and demand of nontoxic and environmentally safe flame retardants. Deflamo has taken its place and strives after growth in this specific market gap.

## **1.1 Aims and objectives**

The aim with this project is to use Deflamo's environmentally friendly FR, Apyrum, together with UPM's new composite material, ForMi GP. Apyrum has not been tested with a cellulose and PP based composite material before so the results could potentially be useful for Deflamo. Fire test and tensile test results of different mixtures of Apyrum and ForMi GP will be compared to find a compound that has good flame retardant properties. The emphasis in this project is on the fire test results and not so much on the mechanical properties of the compounds. Nevertheless suitable processing parameters for the extrusion and injection molding step need to be experimented with in order to get the test specimens made. There will however not be much focus on finding out the optimal production process parameters. The time schedule does not make this possible due to the relatively large amount of different compounds that needs to be made and tested.

## **2. Literature review**

### **2.1 Fire**

To start a fire there needs to be a fuel, oxygen and a heat source that reaches the ignition temperature of the fuel. When the fuel is ignited and starts to burn in the present of oxygen there is enough heat produced to start a chain reaction that feeds itself.

This exothermic chemical chain reaction continues as long as there is enough oxygen, fuel and high enough temperature. If one of these three is removed the fire cannot continue and it dies out.

There are many different polymers that are used together with FRs and they also possess different burning properties. When a polymer burns free O and OH radicals are released that cause the flame spread. A simple figure that explains the combustion process can be seen below in figure 1 (Troitzsch 1990 p. 17).

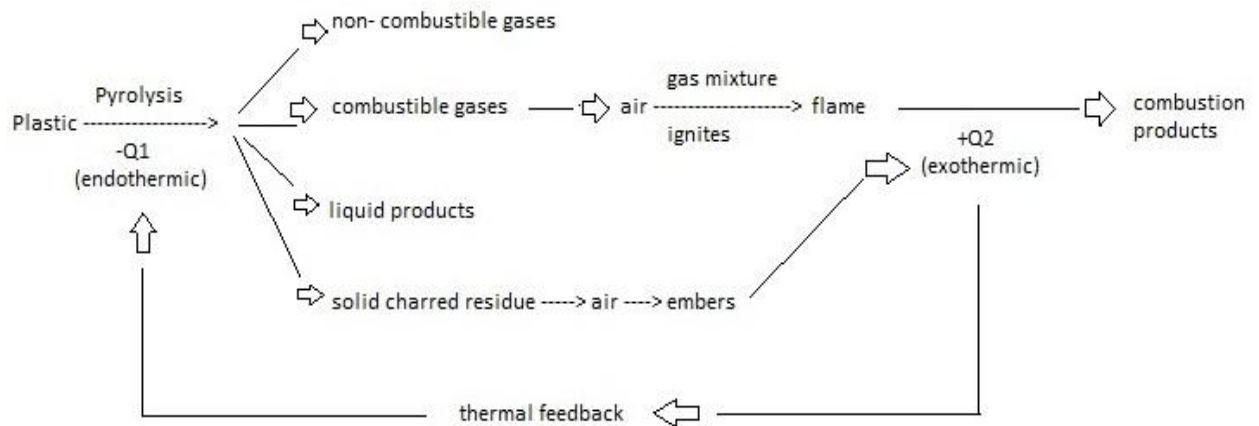


Figure 1. Combustion process. (Troitzsch, *International Plastics Flammability Handbook*. 1990).

## 2.2 Flame retardant function

Flame retardants can function and protect either chemically or physically. Physically acting retardants can act by cooling the fuel to a temperature below that needed for the combustion process to continue. Also a formation of a protective carbon (charring) layer can be used. This layer result in a cooling of the condensed phase and blocks out the oxygen needed for the combustion process (Troitzsch 1990 p. 43). Intumescence also takes place in the condensed phase. In intumescence the burning material forms a layer of char that swells up to foam like substance, which blocks out oxygen and insulates the fuel from the heat source. For Intumescence to work the FR usually needs the following ingredients:

- a catalyst (an inorganic acid) that ramps up the formation of char,
- a charring agent that under combustion is able to form a lot of char,
- a blowing agent that creates a lot of noncombustible gases that causes the char to swell up. (Xanthos 2005 p. 290)

Apyrum is based on intumescence and the blowing agent used in the FR generates only CO<sub>2</sub> and water.

Chemically reacting flame retardants react with the free radical mechanism of the combustion process which take place in the gas phase and thus the fire will eventually be suppressed. The exothermic process is stopped which results in system cooling and in a reduction of flammable gases needed for the combustion process. (Xanthos 2005 p. 291)

### 2.3 Main flame retardants and market

The flame retardant market was valued at approximately 4,2 billion US\$ in 2007 (Reilly & Beard 2009) and a total global usage of approximately 1,8 million metric tonnes (European Commission, Arcadis). According to a market study made by Ceresana the FR market will generate revenues up to 5,8 billion US\$ in 2018. The most widely used FR group are the metal hydroxides that cover 51% of the total consumption. The mostly used single FR is aluminum hydroxide (ATH) and covers 40% of the FR consumption (European Commission, Arcadis). The main flame retardants used can roughly be divided into: halogenated, metal hydroxides and phosphorous containing flame retardants.

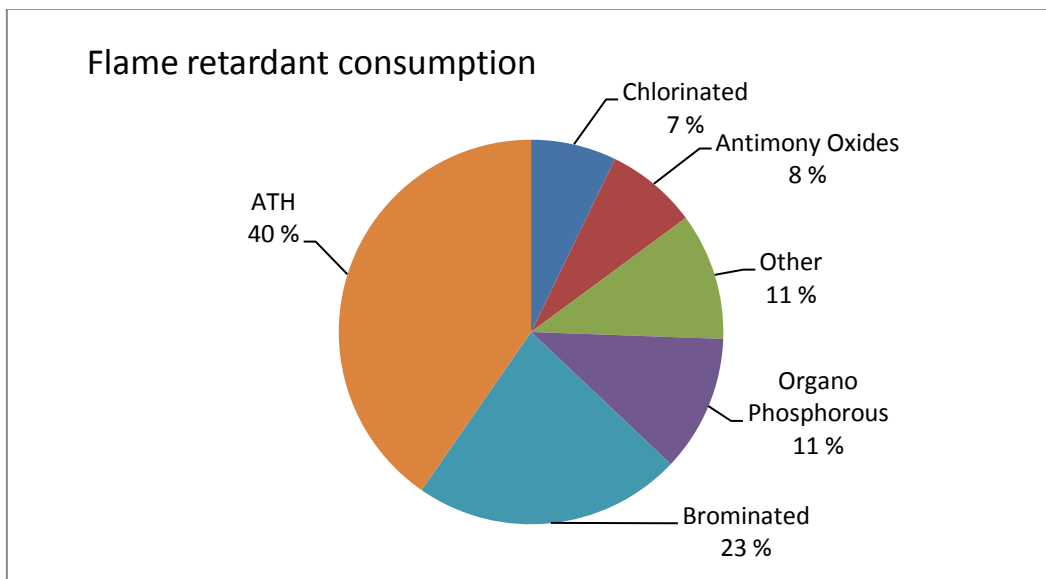


Figure 2. Flame retardant consumption in 2007 .(European commission, flame retardant study).

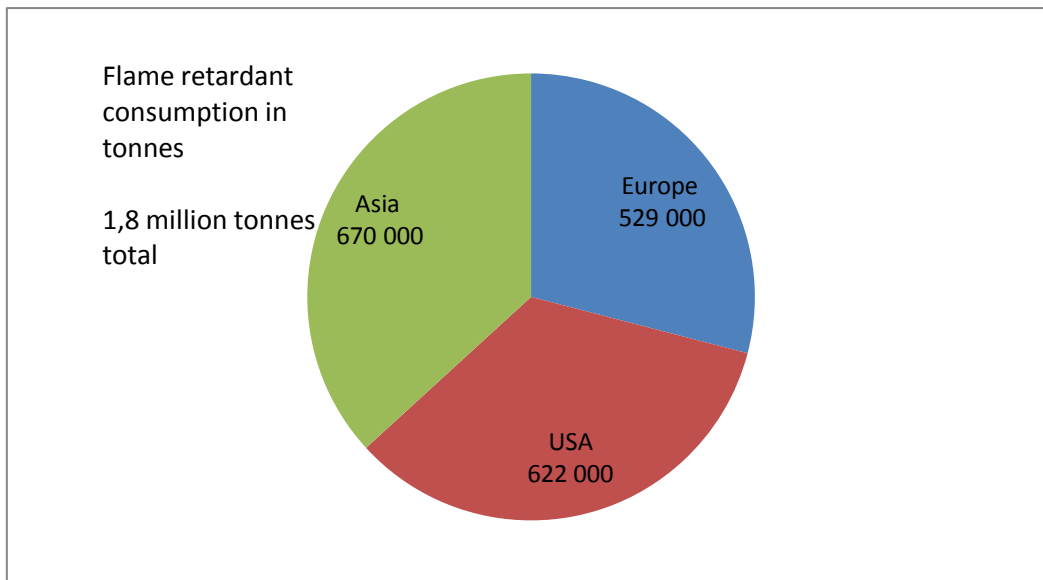


Figure 3. Flame retardant consumption in tonnes in 2007.( European commission, flame retardant study).

### 2.3.1 Inorganic flame retardants

Aluminum hydroxide, ATH ( $\text{Al}(\text{OH})_3$ ) is the most widely used FR. When temperatures exceed  $200^\circ\text{C}$  an endothermic decomposition starts. Here water is released which then dilutes the gases that feeds the combustion reaction. A vapor barrier prevents oxygen from getting in contact with the flame and so it slows down and ends the combustion (Xanthos 2005 p. 293). Magnesium hydroxide is based on the same functions as ATH but have a higher decomposition temperature ( $320^\circ\text{C}$ ) (Xanthos 2005 p. 299).

### 2.3.2 Halogenated flame retardants

Brominated and chlorinated organic FRs act chemically in the gas phase. In the process halogen radicals reacts to form hydrogen halide gas which interferes with the free radical chain mechanism. The high energy radicals are replaced when reacted with hydrogen halide with low energy Br or Cl radicals which are not capable of maintaining the combustion process (Pritchard 1998). According to the European Flame retardant Association (EFRA) there are about 75 different commercial brominated flame retardants on the market. Antimony trioxide is often used as a synergist together with halogenated FRs. PCBs, pentaBDE and hexaBB are examples on halogenated FRs that have been listed as POPs (persistent organic pollutants) by the Stockholm convention.

The Stockholm convention is an international environmental treaty that strives to restrict or eliminate the production and use of POPs.

According to the Brominated Science and Environmental Forum (BSEF) today the main three commercially used BFRs are decaBDE, TBBPA and HBCD. HBCD is at the moment reviewed by the Stockholm convention POPs review committee whether or not it should be added to the POP list and phased out.

### **2.3.3 Phosphorous flame retardants**

Red phosphorous is the most simple phosphorous based flame retardant and is effective in polymers such as polyolefin's, polyamides and thermosetting resins. Other phosphorous based FRs are melamine phosphate, ethylenediamine phosphate and ammonium polyphosphate (APP). APP acts as an intumescent fire retardant system and has been shown to be effective in cellulose based polymers (Troitzsch 1990 p. 47).

### **2.3.4 Other flame retardants**

Other flame retardants are for example melamine, different boron compounds and metal oxides. Nanoclays in combination with e.g. ATH have also been used in polyolefin based cable and wire products (Xanthos 2005 p. 174). Different polymer layered silicate nanocomposites are also widely studied and they show very promising flammability properties. For example a study made by the National Institute of standards and technology (NIST) showed reduced flammability and peak heat release rate for Nylon-6, PS and PP layered silicate nanocomposites. A study by Kashiwagi, Grulke, Hilding, Harris, Awad and Douglas showed promising flame retardant properties in a PP/ carbon nanotube composite.

## **3. EXPERIMENTAL PART**

The flame retardant(s) needs to be incorporated into the composite material to give the final product wanted flame retardant properties. This was done by extruding a mixture containing the chosen components to get new granulates that could be injection molded into the final test specimens. The test specimens were tested in order to find out the flammability and some mechanical properties. Apyrum was compounded on its

own and together with aluminum hydroxide because ATH starts to decompose at lower temperatures than Apyrum. The aim with this was to cover a broader temperature range when the test specimens were set on fire.

### 3.1 Materials

The materials available and used in this thesis project were supplied by Deflamo and UPM. The following raw materials were used,

- ForMi GP(30, 40, 50) granulates
- Borealis bormod™ Bormod-BJ368MO. PP
- Apyrum FR powder
- Martinal OL-104 LEO. Aluminum hydroxide powder.
- Clariant Exolit AP 422. Ammonium polyphosphate powder.



*Figure 4. Borealis virgin polypropylene. Photograph Christian Östman 2012.*

#### 3.1.1 Apyrum flame retardant

As earlier mentioned the Apyrum flame retardant is produced by the Swedish chemical company Deflamo. Apyrum is a 100% environmentally and human friendly flame retardant. It is based on citric acid and all of its individual components are proven food additives. Today it is used in e.g. PVC products and is getting a lot of interest due to its environmental benefits. As earlier mentioned Apyrum acts through intumescence. Apyrum is available as a water based liquid solution or as a fine grained powder. For this project Apyrum in powder form was used and is shown in figure 3.



*Figure 5. From left Apyrum and ATH FR powder. Photograph Christian Östman 2012.*



### 3.1.2 ForMi composite

The new polymer composite material ForMi GP is manufactured by UPM and is made of polypropylene and cellulose. The ForMi GP composite range consists of four variants with cellulose concentrations of 20%, 30%, 40% or 50%. ForMi GP composite is one of three ForMi grades and is suitable for general use for injection molding. The other two grades in the ForMi family are ForMi EFP for thin wall applications and ForMi TP for technical applications.

### 3.2 Mixtures

Following mixtures were successfully (with bold font) extruded and injection molded for further tests. Mixtures with an italic and underlined font were unsuccessfully extruded and only useless products were obtained. GP granulates were compounded with different quantities of Apyrum and ATH to find out the needed weight percentages to achieve wanted flame retardant properties.

Table 1. Extruded mixtures.

	GP30	Apyrum	ATH	Exolit AP422
<b>GP30-1</b>	90%	10%	-	-
<b>GP30-2</b>	80%	20%	-	-
<b>GP30-3</b>	70%	10%	20%	-
<b>GP30-4</b>	100%	-	-	-
<i><u>GP30-5</u></i>	80%	-	-	20%
	GP40	Apyrum	ATH	Exolit AP422
<b>GP40-1</b>	80%	20%	-	-
<b>GP40-2</b>	70%	10%	20%	-
<b>GP40-4</b>	100%	-	-	-
<i><u>GP40-5</u></i>				
	GP50	Apyrum	ATH	Exolit AP422
<b>GP50-1</b>	90%	10%	-	-
<i><u>GP50-2</u></i>	80%	20%	-	-
<i><u>GP50-3</u></i>	70%	20%	10%	-
<b>GP50-4</b>	100%	-	-	-
	Borealis PP	Apyrum	ATH	Exolit AP422

<b>Borealis PP-1</b>	80%	20%		
<u><i>Borealis PP-2</i></u>	70%	10%	20%	
<b>Borealis PP-3</b>	100%			

### 3.3 Processing

The whole processing cycle started with drying and mixing the different components that then would be fed into the extruder. The extrudate containing the additives was then grinded to new granules which could be injection molded. All these steps were repeated for all the different mixtures. It was important to dry and extrude big enough batches of each mixture as both the extrusion and injection molding process produced waste material in the beginning and in the end of the production runs.

#### 3.3.1 Drying

The GP composite granules were dried for at least 3 hours in 115°C to get rid of moisture. The drying temperature and time can be found in the ForMi GP material sheet (appendix, page 40). The Apyrum FR was dried at least for 3 hours in a temperature range between 170-198°C to get rid of moisture. After drying Apyrum weight losses of over 6% were measured. The drying of the Apyrum FR turned out to play a key role in the whole extrusion process. If Apyrum was not dried properly before the extrusion there was water vapor released inside the extruder barrel. This water vapor resulted in an extremely uneven material output and the extrudate was unusable for further processing.

The ready extruded FR containing compound was again dried for 3 hours in 115°C before the injection molding process. The reason for this was that the material was led through a water bath for cooling when it came out of the extruder. Also there were several days between the extrusion and the injection molding processes.

#### 3.3.2 Mixing

When the materials had been dried the GP composite and the FRs were weighed and mixed together in wanted proportions in a closed plastic bucket. The bucket containing the materials was shaken for a couple of minutes in order to mix the different

materials. Visually it seemed that the composite granules and the FR powders stuck to one another relatively good and that this method was good enough to use in the project.

### 3.3.3 Extrusion

The extruder used in this project was a KFM lab ex 18 with a screw L/D ratio of 25/D and a screw diameter of 18mm. It was a small single screw extruder suitable for lab use. The extrusion line can be seen in figure 4.



Figure 6. KFM extrusion line in Arcada. Photograph Christian Östman 2012.

The mixtures containing the GP composites and the FRs were extruded in order to get the final compounds that could be injection molded into the final specimens suitable for testing. Depending on the cellulose and FR contents in the mixtures different processing temperatures and extruder screw RPMs were used. The extrudates had some change in color and were darker than the original granulates. The extruder die used resulted in two continuous solid round extrudate strings. According to the GP composite material sheet the recommended maximum processing temperature is 200°C. The temperatures were kept lower than 200°C in order to avoid or at least minimize thermal degradation of the cellulose fibers and

polymer in the GP composite. The viscosity of the GP composite on its own and also when mixed with the different FRs was relatively high so the lower processing temperatures were kept at around 180°C. The lower temperature limit was used to avoid too much stress on the extruder screw which could result in screw breakage. The RPMs were also chosen to minimize the stress on the screw. Higher RPMs also resulted in some increased degree of “burning” of the material possibly due to higher friction inside the extruder. Also much lower RPMs than what were used for the final specimens made resulted in higher thermal degradation of the extrudate. This could be seen as a color change of the extrudate as shown in figure 5.



*Figure 7. Upper row virgin GP, row below extruded GP. From left GP30, GP40, GP50. Photograph Christian Östman 2012.*

The extrusion parameters used for the final test specimens that were injection molded can be found below in tables 2-4. Many different temperatures (between 175°C and 200°C though) and RPMs were used but the parameters mentioned in this text were the ones that resulted in the steadiest material output and smallest thermal degradation of the material.

### **3.3.3.1 GP30**

Pure GP30 was extruded as well as GP30 containing 10% and 20% Apyrum and 10% Apyrum together with 20% ATH. Different temperatures and RPMs were used

depending on the specific mixture. The extrusion parameters used for the final extrudate are listed below in table 2. From left are the mixtures, RPMs used in the second column and temperatures from the nozzle (left) to feed (right) zone.

Table 2. GP30 extrusion parameters.

100% GP30	40RPM	184°C	182°C	182°C	181°C	180°C	180°C
90% GP30 + 10% Apyrum	32 RPM	182°C	182°C	181°C	181°C	181°C	180°C
80%GP30 + 20% Apyrum	30- 33 RPM	185°C	182°C	181°C	181°C	181°C	181°C
70% GP30 + 10% Apyrum + 20% ATH	25- 32 RPM	181°C	182°C	182°C	181°C	181°C	181°C

### 3.3.3.2 GP40

Extrusion parameters for GP40 mixtures are listed below in table 3. From left are the mixtures, used RPMs in the second column and temperatures from the nozzle (left) to feed (right) zone.

Table 3. GP40 extrusion parameters.

80% GP40+ 20% Apyrum	32 RPM	181°C	181°C	181°C	181°C	180°C	180°C
70% GP40+ 10% Apyrum+ 20% ATH	33 RPM	181°C	180°C	180°C	181°C	181°C	182°C
100% GP40	40-42 RPM	182°C	182°C	181°C	181°C	181°C	180°C

### 3.3.3.3 GP50

GP50 mixture containing 10% Apyrum was extruded successfully. Extrusion parameters for GP50 mixtures are listed below in table 4. From left are the mixtures, RPMs used in the second column and temperatures from the nozzle (left) to feed (right) zone.

Table 4. GP50 extrusion parameters.

100% GP50	37RPM	182°C	182°C	181°C	180°C	180°C	180°C
90% GP50+ 10% Apyrum	37-39 RPM	181°C	181°C	181°C	180°C	180°C	180°C

### 3.3.4 Granulation

The extrudate strings were fed into a granulator at the end of the extrusion line. The machine grinded the FR containing compound into granulates ready to be used for the injection molding process.

### 3.3.5 Injection molding

The injection molding machine used was an Engel 200 ES/ 50 HL CC90 with a clamping force of 500 kN and is shown in figure 6. The test specimens that were injection molded were so called dog bones that are used when determining tensile properties according to the ISO 527-1:1993 standard. Figure 7 shows the 2 cavity mold that was used in the project.



Figure 8. Engel IM machine. Photograph Christian Östman 2012.

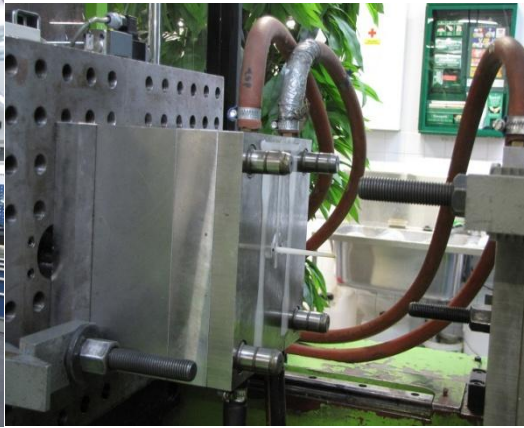


Figure 9. ISO 527-1 dog bone mold. Photograph Christian Östman 2012.

The different compounds were successfully injection molded using process temperatures found on the GP material sheet. The color change after the extrusion step that was mentioned earlier could be seen even more clearly after the injection molding step when comparing 100% GP extruded material with non extruded in figure 8. The final injection molded specimen in figure 9 shows clearly the FR lumps that were formed during the extrusion step. This issue is further analyzed in the next chapter.



Figure 10. From left not extruded, extruded. Photograph Christian Östman 201.



Figure 11. 80% GP30+ 20% Apyrum. Photograph Christian Östman 2012.

As the cellulose and additive content increases in the mixture so does the viscosity. For example pure GP30 that has a cellulose content of 30% has a much lower viscosity than for example GP40 containing 10% Apyrum and 20% ATH. This meant that the injection molding parameters had to be varied to some extent depending on the specific mixture. All compounds were injection molded using the same temperatures, from nozzle to the feeding zone: 195°C, 190°C, 185°C, 180°C. With compounds containing less PP and higher additive contents higher injection speeds, higher holding pressures, higher mold temperatures were used in order to fill the mold cavities. Examples of parameters used for two different compounds is shown in table 5. When pure non extruded GP50 was injection molded higher holding pressure was needed compared to pure once extruded GP50. This could indicate that the cellulose fibers had degraded to some extent during the extrusion step.

Table 5. Example on injection molding parameters used.

	<b>90% GP30+ 10 Apyrum</b>	<b>70% GP40+ 10% Apyrum+ 20% ATH</b>
injections speed(mm/s)	80, 82, 80, 78, 75, 72, 70, 70, 65, 60	75, 80, 85, 80, 78, 72, 72, 68, 60, 55
holding pressure (bar)	50, 55, 55, 60, 55, 55, 50, 50	60, 65, 70, 75, 70, 70, 65, 60
holding time (s)	3	3.5
mold temp.( °C)	65	75
cooling time(s)	22	25
shot size (mm)	43	45
clamping force(kN)	370	420

### 3.4 Processing problems

During the different preparation and processing steps there appeared a number of different complications and issues that needed to be solved. These matters are discussed in the next two chapters



### 3.4.1 Extrusion problems

The moisture content of the Apyrum FR was an essential matter in order to successfully extrude. Too high moisture content in the powder and the extrusion process was impossible to carry out due to the uneven extrusion material output. The uneven material output was seen in very short extrudate strings (the extrudate strings cracked) and material “spitting”. An even spread of the FRs in the GP composite was not achieved in the extrusion process and lumps of FR powders can be seen in the final extrudate in figure 10. The FR powder was also unevenly spread in the two extrudate strings. The extrudate surface was different depending on trough which die nozzle it came out. Also higher FR content could be seen in the extrudate with the more smoother surface shown in figure 11.



*Figure 12. FR lump in extrudate. Photograph Christian Östman 2012.*



*Figure 13. FR lumps and surface smoothness difference in the two extrudate strings. Photograph Christian Östman 2012.*

When extruding the different mixtures gas (water vapor) was formed inside the barrel that resulted in “bubbles” in the extrudate. The effect could be seen in a much

lower density. Below in figure 12 the weight is approximately the same for the non-extruded and for the extruded GP granulates.



*Figure 14. Extruded and non- extruded GP. Photograph Christian Östman 2012.*

80% GP50 and 20% Apyrum could not be extruded successfully due to a very uneven material output and “spitting” of the extrudate. Two trial tests were made and before the second extrusion session it was made sure that the materials were dry but without improvement.

When extruding a mixture of virgin PP and 20% Apyrum the FR powder lumps (agglomerates) gathered in front of the extruder strainer plate causing an increasing pressure. This increased pressure resulted in higher stress on the screw and the ampere meter showed momentary values up to 4,9 amperes. To avoid e.g. breakage of the screw the amperes should be kept under 4,0 as a safety precaution. Two attempts were made and barely enough extrudate needed for the injection molding step was obtained. The true percentage of Apyrum in the extruded PP remained though unknown. It could not be measured how much PP compared to Apyrum came out trough the strainer plate before the process had to be aborted.



*Figure 15. Extruder screw end . Photograph Christian Östman 2012.*



*Figure 16. Clogged strainer plate. Photograph Christian Östman 2012.*

The “burning” or color change of the extrudate was a problem that couldn’t be solved by changing the parameters. When attempting to extrude 80% GP30 and 20% ammonium polyphosphate (Clariant Exolit AP422) the material turned totally black as shown in figure 15. The material stuck inside the extruder barrel and the machine ampere values increased rapidly close to 5. As safety precaution no new attempts compounding GP with Exolit AP422 were made. The temperatures were kept between 180°C- 183°C and RPMs between 8 and 33. Exolit AP422 has a decomposition temperature of >275°C thus should it not start decomposing using the parameters mentioned. One explanation for this phenomenon could be that the true temperatures inside the extruder barrel were much higher due to high friction and shear.

Some possible solutions to these extrusion problems are considered later in chapter 5.1.



*Figure 17. Burnt material, 80% GP30 + 20% Exolit AP422. Photograph Christian Östman 2012.*



*Figure 18. FR lumps in virgin PP. Photograph Christian Östman 2012.*



*Figure 19. Agglomerates in virgin PP. Photograph Christian Östman 2012.*

### **3.4.2 Injection molding problems**

Visually it seemed that the earlier mentioned FR powder lumps or agglomerates are spread in higher volumes in the cavity part that is filled last with help of the holding pressure. E.g. lower holding pressure and larger dosage was used to fix this but the result was not successful. The dosage speeds were also increased to find out if the FR agglomerate problem could be somewhat reduced at this stage, but without any success. Overall the injection molding step was executed without any problems.

## **4. TESTING**

Tensile tests were made in order to find out how the FR powder effects the mechanical properties compared to the pure GP composite. Burning properties were also tested with the different compounds to see the effects on different FR contents used.

### **4.1 Tensile test results**

Tensile tests following the ISO-571 standard were made using so called dog bones as test specimens. The dog bone dimensions are shown in figure 18. The testing machine used was a Testometric M350-5CT and is shown in figure 19. With this mechanical test method results as the modulus of elasticity (stiffness or Young's modulus), elongation (strain) and how much stress (tensile strength) the material can withstand are obtained. Some test results for the different compounds are shown below in tables

six to nine. The results show decreasing tensile strength and strain and increasing values for Young's modulus when increasing the additive contents. The tensile strength and stress values were found to be overall somewhat lower than on the ForMi GP material sheet (appendix, page 40). The FR additives that were used in the compounds resulted in higher stiffness or i.e. Young's modulus. The tensile strength values for the FR containing compounds would presumably have been higher if the FR dispersion in the extruded material had been more uniform and the agglomerate formation problem could have been solved. Figure 20 shows a stress strain curve for GP40 containing 20% Apyrum.

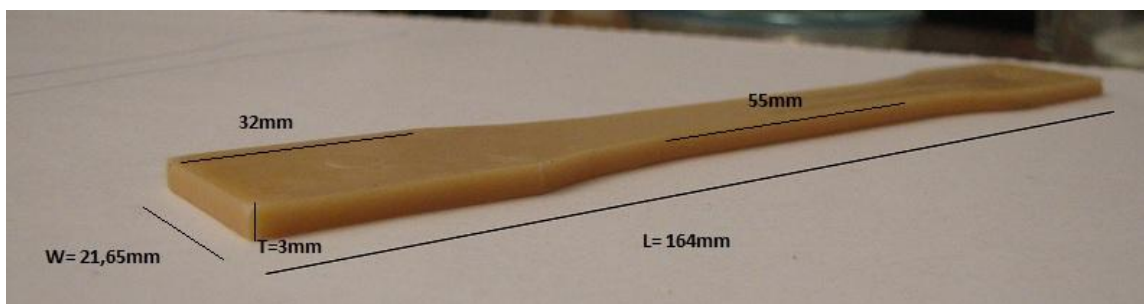


Figure 20. Test specimen. "Dog bone". Photograph Christian Östman 2012.

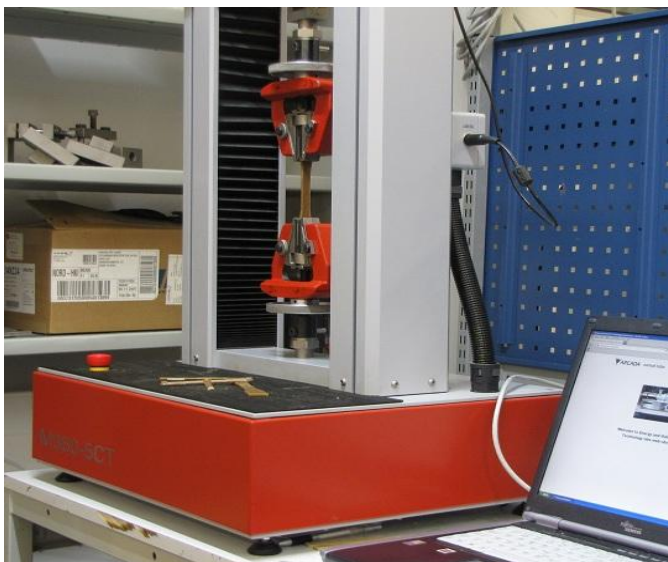


Figure 21. Tensile testing machine. Photograph Christian Östman 2012.

Table 6. GP30 tensile test results.

GP30	100%	100% extruded	10% Apyrum	20% Apyrum	10% apyrum 20% ATH
Stress@Yield, MPa	29.65	26.71	23.68	20.97	21.09
Stress@Peak, MPa	29.65	26.71	23.68	20.97	21.09
Stress@Break, MPa	29.50	26.54	23.45	20.61	20.94
Young's modulus, Mpa	1216	1163	1246	1272.4	1354
Strain@Yield,%	4.495	3.797	3.096	2.668	2.285

Table 7. GP40 tensile test results.

GP40	100%	100% extruded	20% Apyrum	10% Apyrum 20% ATH
Stress@Yield, MPa	37.66	36.97	24.54	23.25
Stress@Peak, MPa	37.66	36.97	24.54	23.25
Stress@Break, MPa	37.56	36.8	24.48	23.2
Young's modulus, Mpa	1310	1288	1388	1526
Strain@Yield,%	5.17	5.138	2.647	2.175

Table 8. GP50 tensile test results.

GP50	100%	100% extruded	10% Apyrum
Stress@Yield, MPa	35.46	30.51	27.23
Stress@Peak, MPa	35.46	30.51	27.23
Stress@Break, MPa	35.37	30.44	27.17
Young's modulus, Mpa	1647	1506	1602.5
Strain@Yield,%	3.456	2.893	2.463

Table 9. PP tensile test results.

PP	100%	100% extruded	20% Apyrum
Stress@Yield, MPa	24.07	24.20	18.22
Stress@Peak, MPa	24.07	24.20	18.22
Stress@Break, MPa	22.68	23.44	17.48
Young's modulus, Mpa	673	688	870
Strain@Yield,%	6.86	6.48	3.36

As mentioned the tensile test results shows decreasing values when comparing them to the GP and PP material sheets. For example the test results gave the tensile stress value 35 MPa for pure non extruded GP50 when the stated value in the material data sheet is 58 MPa. There are some possible reasons for these quite big differences. The different tensile testing equipment used in this project and in tests made by UPM might explain some result differences but is certainly not the main reason. The test specimen and its injection molding parameters might also have resulted the differences. For example the test specimens might have been a bit porous due to too low holding pressures used during the injection molding of the dog bones. The very big differences between the tensile test results and the stiffness values in the material sheet (appendix, page 43) for the Borealis PP could not be explained in this project.

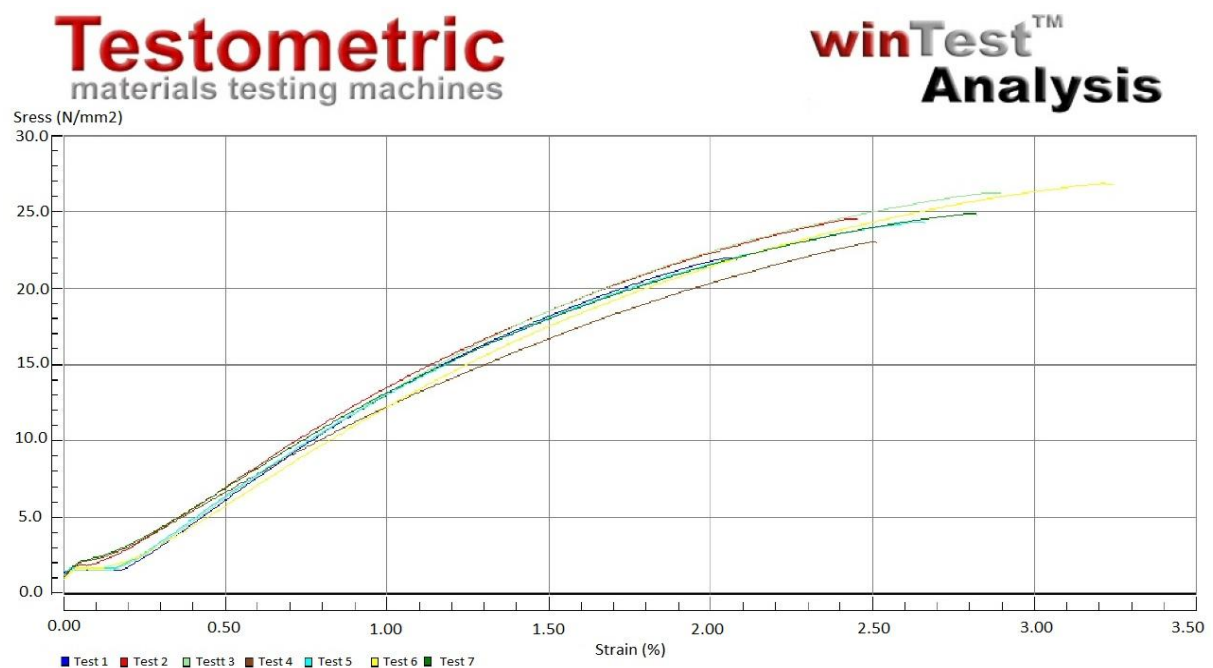


Figure 22. Stress strain curve for 80% GP40+ 20%Apyrum. Photograph Christian Östman 2012.

## 4.2 Burn test results

The burning tests were made in Sweden at Deflammo Ab. The results are not officially comparable to any flammability test standards but do simulate the 50-W horizontal IEC 60695-11-10 test method. All compounds passed both the HB40 (burning rate 40mm/min) and HB75 (burning rate 75mm/min) classes concerning the burning rates. The test

specimens that were injection molded did not have the correct dimensions in order to fulfill the standard criteria's. To achieve these classes the burning rates need to be slower than 40mm and/ or 75mm per minute and no burning material dripping shall occur. All test specimens eventually burnt up and dripping of burning material occurred for all compounds except pure GP50 which reminded of wood like burning (see figures 22 and 23).

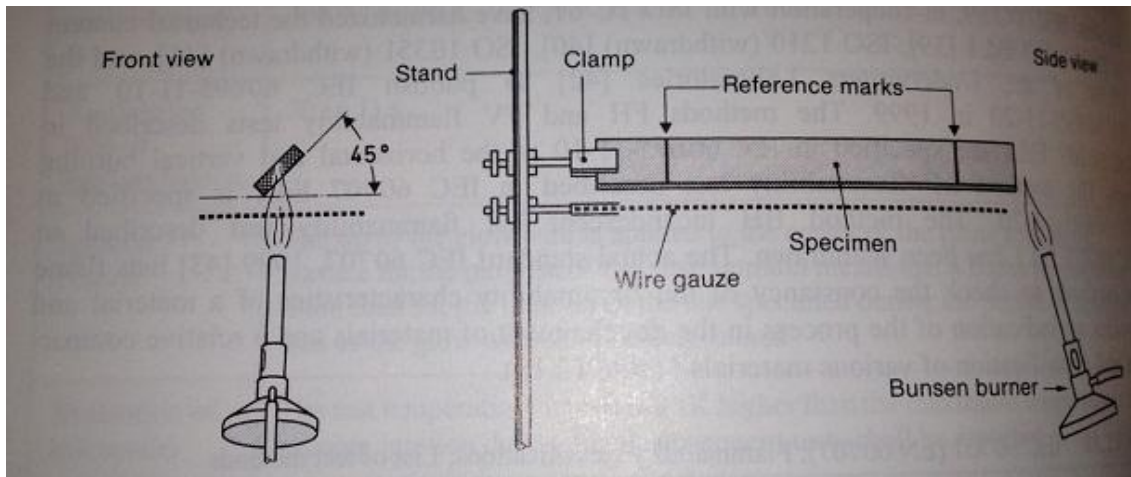


Figure 23. IEC 60695-11-10 Burning test layout. (Troitzsch 1990 p. 356).

The burning rates and test results for the different materials and compounds are shown in tables 10 to 14. Overall some increases in burning time could be measured when using FRs but the reason and function for this remained uncertain. Either the increase of burning time depended on the true FR function or on the dilution of the composite or on both. This question could not be answered during the project. Intumescence caused by Apurym could however be seen during the fire tests as bumps or swellings on the surface compared to a more even surface for 100% GP which is shown in figure 24. The wanted effect would have resulted in a total surface swelling and not in randomly occurring "swellings". As mentioned the test results show increased burning time when the increasing the FR content. The compounds containing 10% Apurym+ 20% ATH gave the longest burning times. Pure GP composite burned faster when it had been extruded once compared to the non extruded. This could be a result of the decreased density (see figure 14) mentioned earlier or by composite fibre degradation that occurred during the extrusion (see color change in figure 10).





*Figure 24. 100% GP50 burns without dripping. Photograph Christian Östman 2012.*



*Figure 25. 80% GP30 20% Apyrum. Material dripping. Photograph Christian Östman 2012.*



*Figure 26. Above GP40 100%, below GP30 20% Apyrum. Photograph Christian Östman 2012.*

Table 10. GP30 burn test 1.

GP30 100%	<i>specimen 1</i>	<i>specimen 2</i>	<i>specimen 3</i>
time/ 100mm	2 min 40 sec	2 min 45 sec	2 min 55 sec
GP30 100% extruded			
time/ 100mm	3 min 25 sec	3 min 20 sec	2 min 46 sec
GP30+ 10% Apyrum 10%			
time/ 100mm	3 min 45 sec	3 min 06 sec	3 in 07 sec
GP30 80%, Apyrum 20%			
time/ 100mm	2 min 58 sec	3 min 22 sec	3 min 59 sec
GP30 70%, Apyrum 10%, ATH 20%			
time/ 100mm	4 min 21 sec	3 min 50 sec	4 min 52 sec

Table 11. GP30 burn test 2.

GP30 100%	<i>specimen 1</i>	<i>specimen 2</i>	<i>specimen 3</i>	<i>specimen 4</i>
time/ 100mm	3 min 20 sec	3 min 55 sec	4 min 12 sec	-
GP30 100% extruded				
time/ 100mm	3 min 12 sec	3 min 10 sec	3 min 30 sec	-
GP30 90%, Apyrum 10%				
time/ 100mm	2 min 40 sec	3 min	3 min 10 sec	2 min 56 sec
GP30 80%, Apyrum 20%				
time/ 100mm	3 min 12 sec	3 min 30 sec	3 min 45 sec	3 min 44 sec
GP30+ 10% Apyrum+ 20% ATH				
time/ 100mm	4 min 50 sec	5 min 18 sec	5 min 5 sec	5 min 40 sec

Table 12. GP40 burn test 1.

GP40 100%	<i>specimen 1</i>	<i>specimen 2</i>	<i>specimen 3</i>	<i>specimen 4</i>
time/ 100mm	3 min 42 sec	3 min 30 sec	3 min 25 sec	3 min 40 sec
GP40 100% extruded				
time/ 100mm	3 min 22 sec	3 min 8 sec	3 min 14 sec	3 min 43 sec
GP40 80%, 20% Apyrum				
time/ 100mm	3 min 20 sec	4 min 30 sec	4 min 48 sec	4 min 23 sec
GP40 70%, Apyrum 10%, ATH 20%				
time/ 100mm	5 min 40 sec	6 min 23 sec	6 min 53 sec	6 min 55 sec

Table 13. GP40 burn test 2.

GP40 100%	<i>specimen 1</i>	<i>specimen 2</i>	<i>specimen 3</i>	<i>specimen 4</i>
time/ 100mm	3 min 20 sec	3 min 47 sec	3 min 40 sec	-
GP40 100% extruded				
time/ 100mm	2 min 42 sec	3 min 18 sec	3 min 42 sec	-
GP40 80%, 20% Apyrum				
time/ 100mm	3 min 49 sec	3 min 45 sec	4 min 44 sec	4 min 58 sec
GP40 70%, Apyrum 10%, ATH 20%				
time/ 100mm	5 min 5 sec	4 min 27 sec	7 min 6 sec	5 min 56 sec

Table 14. GP50 and PP burn test.

GP50 100%	<i>specimen 1</i>	<i>specimen 2</i>	<i>specimen 3</i>	<i>specimen 4</i>
time/ 100mm	4 min	4 min 5 sec	4 min 51 sec	-
GP50 100% extruded				
time/ 100mm	3 min 20 sec	4 min 30 sec	3 min 50 sec	-
GP50 90%, Apyrum 10%				
time/ 100mm	5 min 45 sec	5 min 55 sec	5 min 20 sec	5 min 11 sec
PP 100%				
time/ 100mm	2 min 30 sec	4 min 5 sec	2 min 55 sec	-
PP 20% Apyrum				
time/ 100mm	4 min 25 sec	6 min 18 sec	3 min 11	-

## 5. DISCUSSION

As new green and more environmentally friendly products and materials have gained interest among consumers as well as producers the demand and need for new environmentally friendly flame retardants also grow. If a product can be made to be environmentally friendly as well as cost efficient it has good potential to attract new customers. Also with the increasing concern around the halogenated flame retardants and their health effects new possibilities arise.

Further trial tests would need to be made with proper equipment and experience in order to find out the true fire retardant properties of Apyrum in the GP composite.

## 5.1 Extrusion issues and possible solutions

The main issue in this project was the earlier mentioned agglomeration of the flame retardant powders during the extrusion process. This could not be solved during the project and using the equipment available. This issue also resulted negatively in the FR properties of the final product. The agglomerate formation mechanism in single screw extruders is shown in figure 27.

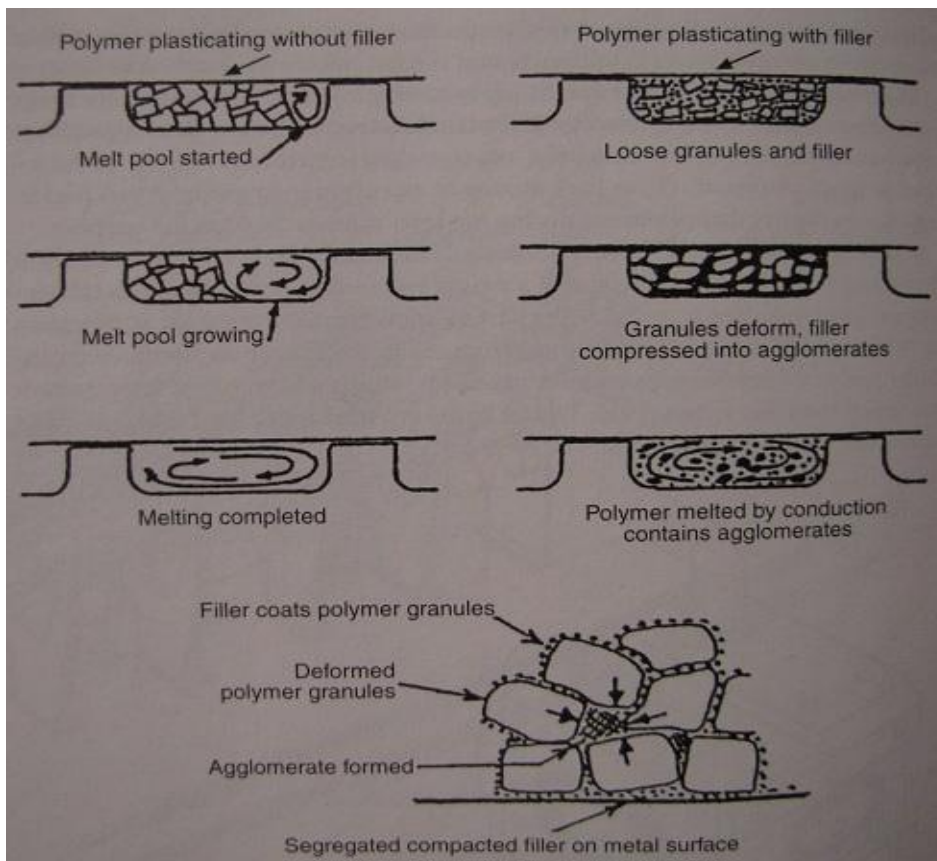


Figure 27. Mechanism of agglomeration formation. (Xanthos 2005 p.46)

The compounding of Apyrum and the ForMi GP composite might need a twin-screw extruder and/ or a screw that enhances mixing. In the extrusion process two different mixing types happen, distributive and dispersive mixing. Distributive low shear mixing, distributes the particles evenly in the melt while dispersion mixing uses high shear to break up e.g. agglomerates and disperses the particles evenly throughout the melt. Dispersion mixing is used e.g. when mixing flame retardants and lubricants. One way to prevent agglomeration formation is to feed the filler or flame retardant

downstream when the polymer is fully melted in the extruder barrel. This could not be done with the extruder used in this project. For enhanced mixing, screws with mixing sections, co- or counter rotating twin- screw extruders, gear mixers and kneading blocks can be used. (Giles *et. al.* 2005 p. 43, 120). Figure 25 shows some examples on mixing sections for a single screw extruder and figure 26 shows examples of kneading blocks.

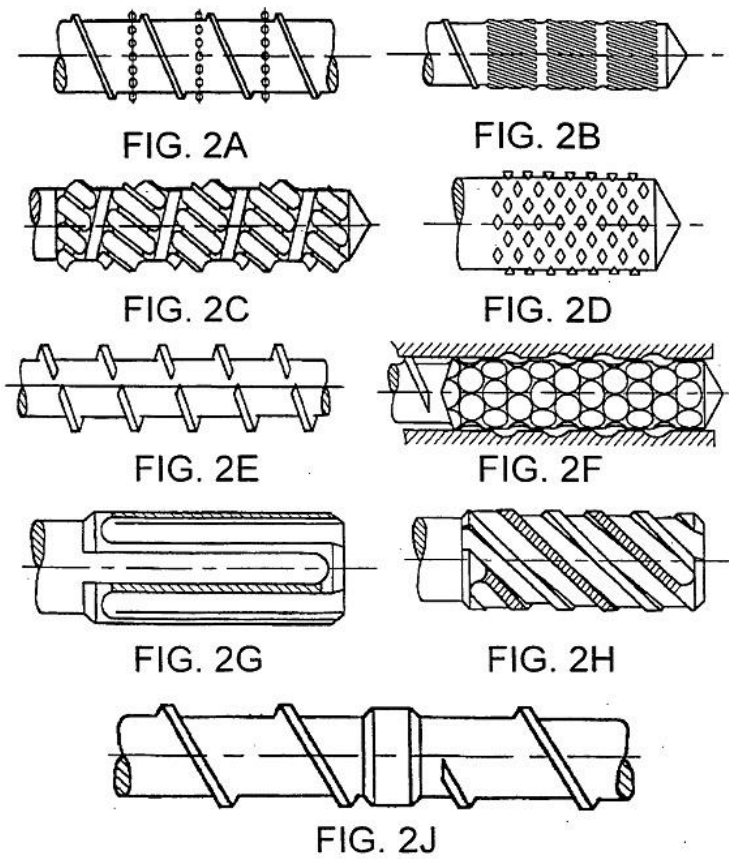


FIGURE 2 A-J Prior Art

Figure 28. Patented dispersion and distributive mixing sections. Rauwendaal Extrusion Engineering Inc.



Figure 29. Examples of kneading blocks for a twin-screw extruder from the German company Extricom.

Lubricants are used for example when reduction in melt viscosity and friction is needed. Coupling agents can also be used to increase the interfacial bonding between the matrix and additive filler (Xanthos 2005 p. 339). The gas formation inside the barrel resulted in pressure increase and bubbles in the extruded material and could possibly be solved using an extruder with a vent that let volatiles and moisture to escape (Giles *et. al.* 2005 p. 47).

## 6. CONCLUSION

The results acquired did not correspond to those that were set up when starting this project. However the different practical and production steps were executed somewhat successfully and it was shown that there is a good possibility that Apyrum could be compounded with the GP composite using an extruder. This kind of project might need some extrusion consulting and more knowledge regarding compounding. The extrusion process was relatively demanding due to the lack of extruding experience and the new materials and additives used. The injection molding of all compounds made was executed without any problems when not taking the agglomeration issue from the extrusion step into consideration.

The number of different compounds made in this project as well as the problems during the extrusion step resulted in a lack of time used to find optimal processing parameters for the materials and to solve the agglomeration problem. During this project approximately a total of 80 specimens were fire tested and about 90

specimens tested with the tensile testing machine to get the results that are stated in this document. 7 different FR containing compounds were made and tested. If there would have been more time more compounds would have been made for comparison. The project taught a lot about the extrusion process and its potential complications as well as material testing.

There are many scientific studies made involving flame retardants, especially BRFs and their impact on humans and the environment. The down side is that many of these publications cost money. This means that the number of scientific studies concerning recent studies used in this project is limited. ScienceDirect is an example of an online database where many articles and studies on flame retardants can be found. The main concerns and trends regarding BRFs were though stated in this project.

The project and its content can be useful for Deflamo in the future for example in problem solving concerning the customers extrusion processes. Also it needs to be pointed out once again that Apyrum had not been compounded with the ForMi GP or with virgin PP before. For this reason alone it was very interesting and useful to do this project.

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## 8. APPENDICES

UPM ForMi GP. Technical datasheet:

[http://www.upm.com/EN/PRODUCTS/composites/upm-composite/Documents/UPM\\_ForMiGP.PDF](http://www.upm.com/EN/PRODUCTS/composites/upm-composite/Documents/UPM_ForMiGP.PDF)

Martinal OL-104 FR powder. Aluminum hydroxide. Technical datasheet:

[http://www.martinswerk.com/allproducts\\_e.aspx](http://www.martinswerk.com/allproducts_e.aspx)

Borealis bormod™ Bormod-BJ368MO. Technical datasheet:

[http://www.borealisgroup.com/pdf/literature/borealis-borouge/product-news/K\\_PNN067\\_GB\\_MO\\_2007\\_10\\_BB.pdf](http://www.borealisgroup.com/pdf/literature/borealis-borouge/product-news/K_PNN067_GB_MO_2007_10_BB.pdf)

All sites above accessed 24.11.2012.

# Technical Specification UPM ForMi GP

11/11/2011

**MATERIAL** UPM ForMi is new cellulose fiber reinforced plastic composite with high renewable material content. It is specially designed for injection moulding applications. Principal ingredients are specially selected cellulose fibers and virgin polypropylene. Cellulose fibres significantly increase stiffness and strength of polypropylene.

**APPLICATIONS** Environmentally sound UPM ForMi composite can be used in injection moulding applications instead of polypropylene, filled polypropylene or several other plastics.

**ENVIRONMENT** UPM ForMi is manufactured from renewable cellulose fibers which reduce usage of oil based plastics. Material is fully recyclable or can be burned for energy. All cellulose fibres are from sustainably managed forests.

**PHYSICAL AND MECHANICAL PROPERTIES**

Property	Test method	GP 30	GP 40	GP 50
Density, g/cm <sup>3</sup>	ISO 1183	1.02	1.07	1.12
Tensile strength, N/mm <sup>2</sup>	ISO 527-2	41	50	58
Tensile modulus N/mm <sup>2</sup>	ISO 527-2	2900	3800	4700
Strain (tensile), %	ISO 527-2	4.8	4	3
Charpy impact strength, notched, kJ/m <sup>2</sup>	ISO 179/1eA	4.2	5.5	3.7
Charpy impact strength, unnotched, kJ/m <sup>2</sup>	ISO 179/1eU	34	45	29
Cellulose content, weight %		30	40	50

**PRETREATMENT** UPM ForMi granulates are ready to use, but it is recommended to dry granulates before injection moulding. Recommended drying temperature and time is 115 °C and 3 hours.

**INJECTION MOULDING** UPM ForMi does not need special equipment for processing. Recommended processing parameters for typical injection moulding machine are:

Temperature profile from nozzle	195/190/185/180 °C
Injection pressure	<1200 bar
Mould temperature	+60 - +80 °C
Injection speed	As high as possible

**SAFETY** Maximum recommended processing temperature is 200 °C. Overheating may cause risk for the thermal degradation. Auto-ignition of UPM ForMi material is possible after purging the injection moulding machine.

**STORAGE** UPM ForMi granulates should be protected from UV-light and stored in closed packages in dry conditions at temperature below 50 °C. Air humidity can increase moisture content of the material and have negative effects on the end product properties.

**Einsatzbeispiele /  
Applications**

MARTINAL OL-104 LEO und OL-107 LEO sind neue, feinteilig gefällte Aluminiumhydroxide mit verbessertem Compoundierverhalten, höherer Temperaturstabilität, verbessertem Fließverhalten und höherem Schüttgewicht nach Förderung sowie niedrigerer Compoundviskosität. Zielpolymere sind Polyolefine und deren Copolymere, PVC und Elastomere.

MARTINAL OL-104 LEO and OL-107 LEO are new fine precipitated aluminium hydroxide grades with improved compounding performance, higher thermal stability, better powder flowability and higher bulk density after conveying, as well as lower compound viscosity. Target polymers are polyolefins and their copolymers, PVC and Rubber.

**Durchschnittsanalyse /  
Typical analysis**

		OL-104 LEO	OL-107 LEO
Al(OH) <sub>3</sub> -Gehalt /-content	[%]	= 99.4	= 99.4
Feuchte / Moisture (105 °C)	[%]	≤ 0.35	≤ 0.4
Glühverlust / Loss on Ignition (1200 °C)	[%]	= 34.5	= 34.5
Na <sub>2</sub> O löslich/soluble	[%]	≤ 0.08	≤ 0.08
Refractive Index	[%]	1.58	1.58
Teilchengröße (Laserbeugung, Cilas) / Particle Size (Laser scattering, Cilas) d <sub>50</sub>	[µm]	1.7 - 2.1	1.6 - 1.9
Spezifische Oberfläche / Specific Surface Area (BET)	[m <sup>2</sup> /g]	3 - 5	6 - 8
Ölaufnahme / Oil absorption	[ml/100g]	27 - 32	28 - 33
Elektrische Leitfähigkeit / Electrical conductivity (10 % in H <sub>2</sub> O)	[µS/cm]	≤ 60	≤ 70
Weißgrad / Whiteness	[%]	≥ 94.0	≥ 94.0
Schüttdichte / Bulk Density	[kg/m <sup>3</sup> ]	= 400	= 350
Dichte / Density	[g/cm <sup>3</sup> ]	2.4	2.4
Wärmebeständigkeit / Thermal Stability (Temp. at 2% weight loss, 1 K/min)	[°C]	= 225	= 220



### Shipping Information

Transportation classification: not regulated for transportation  
Harmonized tariff number: 28183000

Packaging and minimum order quantity information is available from sales or customer service.

### Safety and Handling Information

For specific safety, toxicity and handling information, please refer to the material safety data sheet on this product.

### Chemical Registration Numbers

Chem. Bezeichnung / Chem name: Aluminium hydroxide  
Formel / formula:  $\text{Al}(\text{OH})_3$   
CAS No.: 21645-51-2  
EG/EINECS No.: 244-492-7



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Issue: October 2007  
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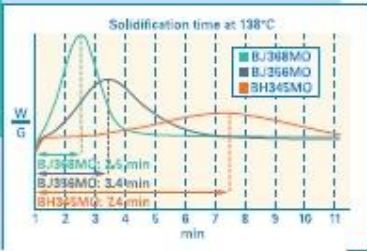


Figure 1: Top productivity with instant solidification: Borealis Nucleation Technology makes BJ368MO solidify faster, allowing a significant reduction in cycle time

## BNT Nucleation for high productivity

Borealis Nucleation Technology (BNT) is state of the art in nucleation technology.

- In situ (in reactor) nucleation
- Eliminates plate out and liability issues relating to nucleating additives
- Very fast crystallisation and production cycle potential

## Bormod BJ368MO brings impact and stiffness at a high flow rate

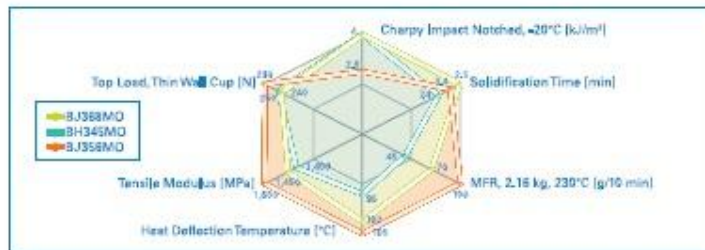


Figure 2: Property profile of BJ368MO vs high impact and high flow references

	Unit	BJ368MO	BH345MO	BJ356MO
MFR, 2.16 kg, 230°C	g/10 min	70	45	100
Tensile modulus	MPa	1,450	1,400	1,600
Stress at yield	MPa	26	26	29
Strain at yield	%	4	5	4
Charpy 23°C/-20°C	kJ/m <sup>2</sup>	5.5/4	6/4	4.5/2.5
Instrumented falling weight, 0°C/-20°C	J	14/11	30/22	10/10
HDT 0.45 MPa	°C	102	95	105
Crystallisation temperature	°C	131	124	128
Drop test, -20°C, Thin Wall Cup, 850 ml glycol	m	2	2.5	-
Top load, Thin Wall Cup	N	240	250	290
Shrinkage, flow/cross direction	%	1.66/1.68	1.64/1.62	1.67/1.68

K: FN No. 01705 MO 2007 10 08

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