Project No: *516225*

Project Acronym: FLOWFREE

The development of a high output processing method for the extrusion of solid thermoplastic sheet and profile.

Instrument: Collective Research

Thematic Priority: Frame Work 6

Final

Activity Report

Start date of project: 1st September 2005 (Duration: 3 Years)

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Project coordinator: Dr Gary Foster (Smithers Rapra Technology)

Revision: Final version

Project co-funded by the European Commission within the Sixth Framework Programme (2002-2006)		
Dissemination Level		
PU	Public	✓
PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	

Contents

Project Overview

Summary description of project objectives

A technology developed by two of the consortium partners has shown that plastics extrusion equipment can be modified to allow for compressed CO_2 to be used as a processing aid for the production of solid extrudate with reduced melt viscosities, thus allowing processing at faster output rates, reduced temperatures or reduced energy.

The main objectives of the project were to:

Develop a prototype extruder that will allow full industrial, commercial development of the process.

Develop the process on an industrial scale for SMEs with different production requirements.

Demonstrate the technical and commercial viability of the process on full industrial scale.

Carry out dissemination and training activities that allow for maximum exploitation of the technology.

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Contractors involved

N°	**Participant name**	Short name	Country
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3	European Plastics Converters	EuPC	EU
4	Plastic Machines Promotions	Prom	IT
5	Asociacion Valenciana De Empresarios De Plastico	AVEP	ESP
6	Turkish Plastics Industry Association	PAGEV	TUR
8	Plastire	Pla	ESP
9	Reboca	Reb	ESP
10	Yelkeniciler	Yel	TUR
11	AGOR	Rog	GER
12	Cesap	Ces	IT
13	Presearch	Pre	UK
14	British Plastics Federation	BPF	UK
15	Aimplas	Aim	ESP
16	Queens University Belfast	QUB	UK

Technical Execution

Introduction

This project considered the use of CO_2, in a supercritical state (sc), as a processing aid to overcome typical high viscosity problems encountered during melt extrusion and to increase processing efficiency and reduce costs. Although CO_2 is commonly used to foam polymers, the objective was to develop a new technology that enables the production of solid extrudate, with the associated benefits of reduced melt viscosity, thereby potentially allowing processing at faster output rates, lower temperatures and with reduced energy consumption.

Prototype systems were initially tested at three Research partners, parameters optimised, and then scaled-up and installed at four industrial locations to enable testing of the process within a production environment. IPR developed during this process was protected for future exploitation by consortium members.

Training materials, a website, and a best practice guide were produced to aid dissemination of the project findings and to encourage take-up by industry.

Preliminary Work

A literature survey was undertaken to establish the current state-of-the-art, and a review conducted of existing viscosity modifiers to establish criteria for comparison with results using $scCO_2$. The different viscosity modifiers identified were tested to quantify changes in output and energy consumption.

The general requirements for the extrusion equipment, and the initial materials to be tested were also defined. This was done in conjunction with the industrial partners to ensure that the materials tested matched the normal range of materials in use on their production plants.

Development of Prototypes

The initial stage of this project focused on the prototype extruder development, at Queens University, Belfast, and at Aimplas, Valencia, Spain. The aim was to establish a continuous system that would allow the injection of super critical CO_2 into polymer melts to produce a compact, *foam-free* extrudate. Product characterisation and further process optimisation followed to understand the full effect of the CO_2 on the material processed.

The extruder was modified to allow for incorporation of CO_2 during melt processing. The technology consisted of a dual syringe pump, a Back-Pressure Regulator (BPR), and an injection valve, and was able to deliver and maintain a stable CO_2 delivery during polymer extrusion. A melt seal needed to be created before CO_2 injection to prevent back leakage flow of CO_2 toward the hopper. The basic system is shown in Figure 1.

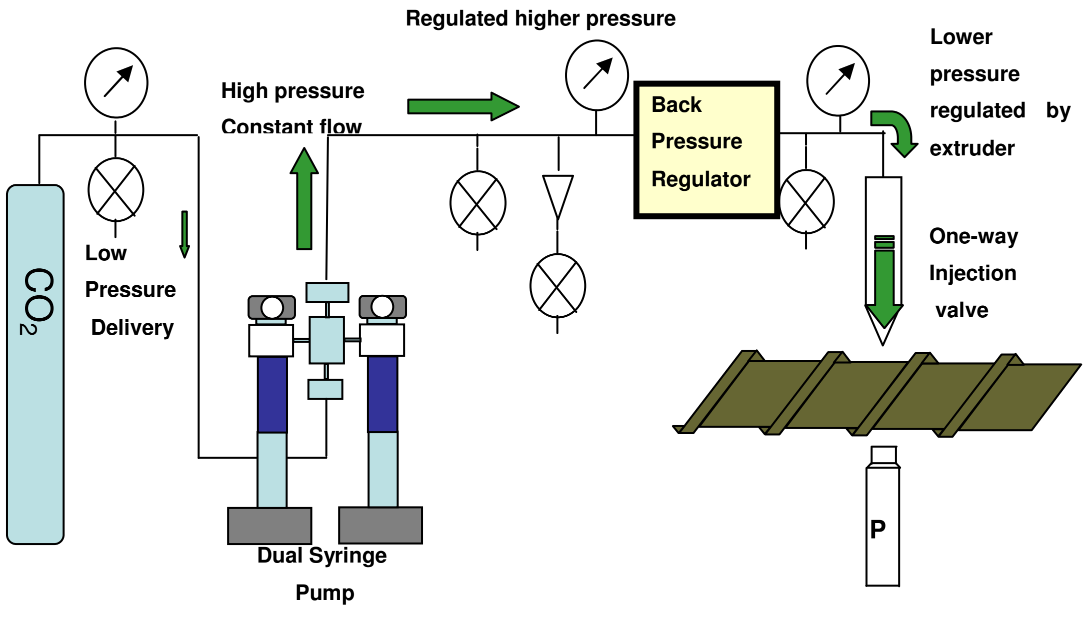

Figure 1: CO_2 delivery technology developed during the study.

For the preliminary experiments, various polymers were chosen to demonstrate the plasticising effect of CO_2 using the development system. Extruder pressure at the end of the screw (P3) was monitored as an indication of the change in viscosity. Reduction in extruder motor power, which indicated higher energy saving, due to the CO_2 plasticising effect, was also observed.

As the CO_2 plasticised the polymer, the benefits observed included reductions in processing pressure, reductions in motor current, and an increase in extruder output rate.

Using rigid PVC was seen to give greater benefits than with other polymers so a sample was obtained from one of the consortium partners. The technology was then applied during the melt extrusion and it was shown that, the more CO_2 added the greater the benefits observed.

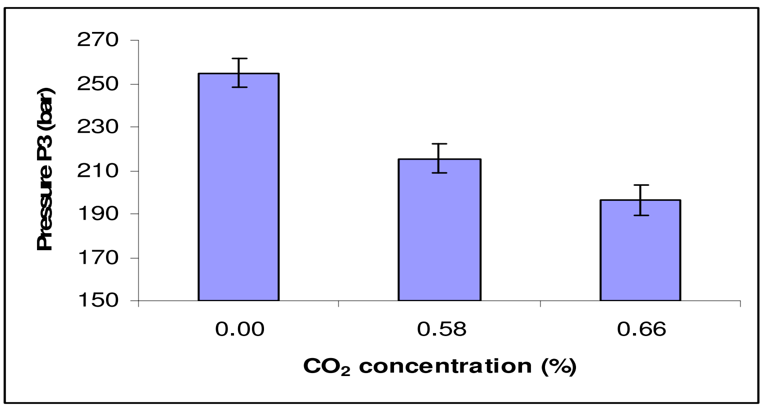

Figure 2: The effect of CO_2 levels on die pressure for rigid PVC extrusion

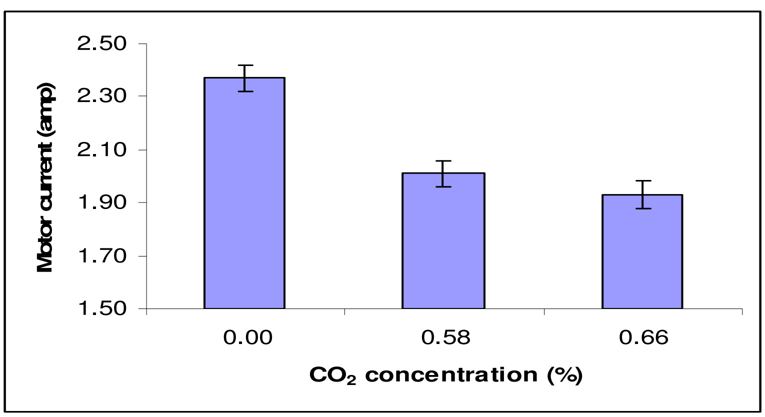

Figure 3: The effect of CO_2 levels on motor current for rigid PVC extrusion.

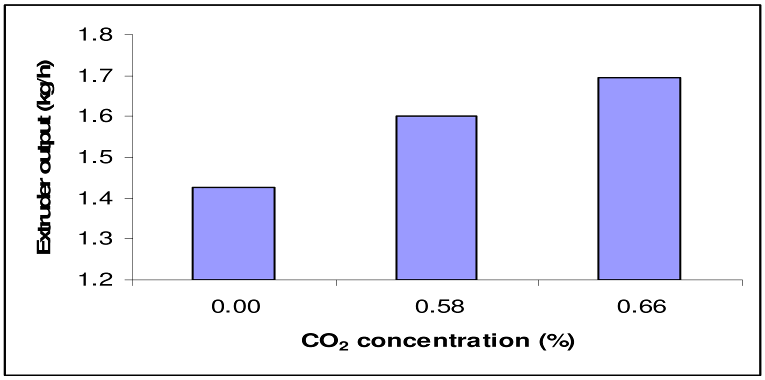

Figure 4: The effect of CO_2 levels on output rate for rigid PVC extrusion.

Optimization of Prototypes

The prototypes continued to be optimised throughout the duration of the project studying different materials, CO_2 injection levels, injection position along the barrel, screw design, and die design. New findings were fed through to the industrial installations to assist with the take-up of the technology within industry. Some of the key points relating to the optimisation of the prototypes follow;

Design of experiment

The Design of Experiments (DOE) technique was used in order to quantify the effects of different extrusion parameters, especially in relation to foaming of the extrudate. Various processing conditions, as well as extruder design factors were all found to have a significant influence on the performance of the process. Additionally it was noted that higher pressure resulted in more CO_2 being dissolved and diffused in uPVC with greater plasticising benefits. Hence die design, and process temperature, were also important to CO_2 absorption. Also the CO_2 had to be well mixed into the polymer as poor mixing and distribution resulted in poor product quality.

Screw design

A new PVC double-flighted screw, based upon a normal PVC screw, but with different compression and metering zones, was tested to determine how the screw design parameters affected the process.

By altering the pressure profile in the barrel, and creating a more effective melt seal, CO_2 is prevented from flowing back. The shortened compression and longer metering resulted in higher CO_2 absorption and diffusivity giving higher plasticising benefits.

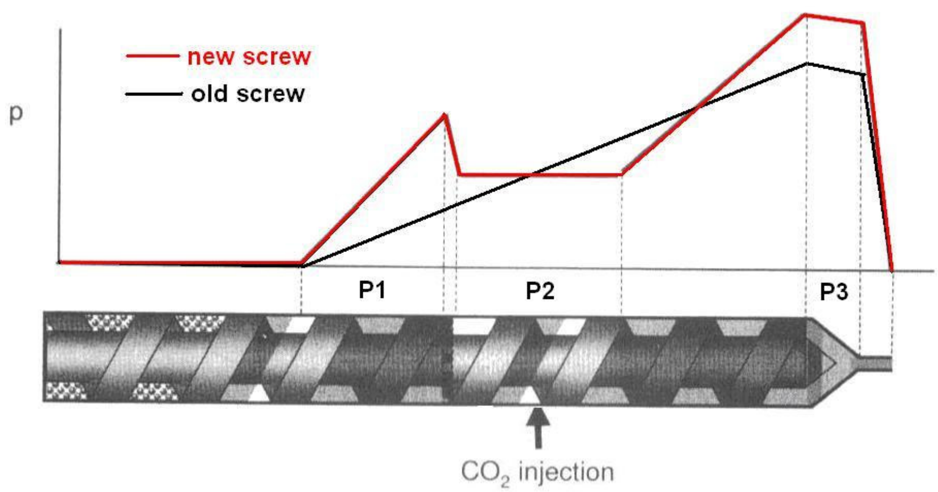

Figure 5: Comparison of pressure profiles between new and old screws

Die design

Extensive optimisation of the die design at QUB has shown that improved die designs can significantly reduce the foaming effect and move closer to solid extrudate and reduced energy.

The Mk 2 die showed a remarkable difference in the potential processing benefits of extrusion with uPVC. In particular the reduction in specific energy was improved from 6% to 30%. Additionally the quality of the extruded surface was improved as shown under scanning electron microscopy (SEM) where no voids could be identified when using the Mk 2 die.

The final Mk 4 die enabled better control to be exerted over pressure drop and CO_2 addition. It had an adjustable geometry thus allowing better control of the process within the die.

Throughout the project emphasis has been placed on the design of the die and this has progressed from the original Mk1 die with a 2mm slot, via the Mk II with improved temperature control, eventually leading to the Mk IV which has the design capability to vary its geometry.

In summary, the appearance and growth of voids and bubbles can be limited or prevented by carefully controlling aspects of the extrusion process. For rigid PVC processing, only a slight change in conditions was necessary to prevent the appearance of void/bubbles, especially on the surface of the extrudate.

<u>IPR</u>

All of the findings lead to the conclusion that the process offers real advantages for PVC extrusion. Patentable technology has been applied to the complete CO_2 assisted extrusion process in order to achieve maximum processing benefits. Arrangements are in place to actively seek exploitation within the extrusion sector of the plastics industry.

Effect of CO_2 on Polymer properties

It was found that the properties of the materials produced with CO_2-assisted technology were similar to those obtained using the conventional process, without CO_2 assistance.

A study was carried out, as part of the PhD which was integrated within this project, using an off-line high pressure chamber. This proved to be an effective technique to study the interactions between CO_2 and solid polymer. It was found that no foaming was observed during the studies and after diffusion of CO_2, from the polymer, there were no significant changes in the properties of materials processed with and without CO_2.

A range of characterisation techniques was employed to investigate the properties of the materials produced with the CO_2-assisted technology. These include DMA, XRD, IR spectroscopy, SEM, mechanical tests and density. There were no significant changes observed in the properties of materials processed with and without CO_2. This means that CO_2 could be used as a temporary plasticiser during processing, giving benefits of significant reductions in viscosity and extruder motor power and an increase in material output, without causing significant changes in the properties of the materials.

Sorption and desorption studies were carried out. Absorption time was found to be the most influential parameters in CO_2 sorption, even though elevated temperature and pressure could be used in some cases to shorten the absorption time. With the presence of CO_2, the plasticisation effects were clearly observed. As the CO_2 diffused out of polymer, these effects diminished and the materials retained the original properties.

Scale up to Industrial Installations

The Industrial installations at each SME were commissioned and tested with all researchers giving technical support at one or more of the SMEs.

Each installation was different, with different materials or end product being run, but each SME was looking for a commercial opportunity to use compressed CO_2 as a processing aid.

The scale-up to the industrial installations commenced with the definition of the equipment modifications necessary, for adapting the CO_2 pump system, to each individual extrusion line selected for the industrial trials. The extruder size and the particular screw configuration were the basis for deciding the position of the injection points as well as the number and position of temperature and pressure transducers. All these changes in the extruder configuration took an important part of the planned time as it implied also modifications in the extruder barrels.

Support was provided during the pump installation and the industrial trials themselves. Optimization of the extrusion parameters, according to each process, and the different materials tested, was carried out. The materials tested ranged from random PP and LDPE extruded pipes, PS strip, TPE, and rigid PVC tubes. Different levels of CO_2 were tried in all cases. It was also observed that, depending on the material, the processing benefits like torque reduction or output increase were often very different.

Economic Viability

One objective was to evaluate the technical, environmental and economic performance of the system. Hence, a chart was prepared showing the pay-back times for different investment scenarios with the conclusion that in certain circumstances pay-back could be achieved within an acceptable time scale. This was dependent upon a variety of factors including both the scale of operation and the polymer type being processed. It was found that PVC showed the greatest reduction in specific energy, when using the FlowFree process, so a separate pay-back chart was constructed specifically covering PVC. This showed PVC to be the most cost effective operation to benefit from the technology.

Best Practice Guide

By collating all the relevant research, and the details learnt from the industrial installations, it was possible to draw up a 'Best Practice Guide' for use within the consortium. This covered the materials that would most benefit from the process, the ideal set-up, techniques of CO_2 injection, and ideal designs for the extruder screw, barrel, and die. This forms the basis of the patent applied for within the project.

Training and dissemination

One of the important aspects of this type of research project is to ensure that knowledge, that will benefit the EU industrial community, is made widely available in a clear and understandable way. Hence the main partners responsible for dissemination were the IAGs who, by their very nature, are used to ensuring that their membership are kept up to date with all new developments in the industry. Their SME membership was therefore continually updated by means of regular newsletters, either digitally or by hard copy.

The RTDs task was to consolidate the raw scientific findings into a format that could be most readily assimilated by the general community. Hence the RTDs produced the basic training material, fliers, and posters that were used as the main sources of dissemination material. However, the RTDs first needed to conduct a scientific study, understand the science, and produce a top level review of the research. This can be found in the PhD thesis submitted by Febe Kusmanto of QUB, and the paper presented at the ANTEC conference in Milwaukee, WI, USA in May 2008. This enabled us to disseminate the knowledge within the scientific community as well.

Finally we needed to ensure that the wider community, outside of the scientific grouping and the IAG membership, were also covered. This was acheived by targeting shows and exhibitions that were open to the

general public where poster displays and leaflets could be used to target the wider audience.
Hence we were able to cover the scientific community, the large group of SME members of the different IAGs, and the general public, including the plastics industry as a whole.

During the project a full range of training materials were developed, which covered all aspects of the process. They took the form of Powerpoint presentations and included the following topics:

- Training Overview
- Supercritical CO_2
- Alternative Process Modifiers
- QUB System
- Process Benefits
- Economic Assessment
- Technology Requirements
- Industrial Scale-up

The training materials are available for download from the public part of the website.
A Trainers' Workshop was organised in order to train project trainers from the IAGs and technical staff and took the form of a virtual workshop, with the training materials being available on a CD-Rom and accessible via the project website.

A training session was also held towards the end of the project, at EuPC in Brussels, to provide information to a wide range of SMEs. This workshop took the form of a formal, classroom style training event, where some of the principal researchers on the project gave presentations covering the technical work undertaken, and also the project results. This event had approximately 30 attendees. In addition to the formal training, attendees were also provided with a FlowFree information pack, which contained useful leaflets and flyers about the project, as well as the Training Materials CD-Rom.

Finally, a FlowFree Training Materials CD-Rom was produced, to bring together all of the project training materials as well as other useful information, into one user-friendly, easily navigated package. The CD-Rom contains the following information:

Project Flyer

Project Posters

Training Presentations

Research Paper

This Training Package is also available on the FlowFree website for members of the public to download.

FlowFree has been promoted, both to IAG SME members and external members of the public via a range of promotional activities, such as the submission of articles to the relevant trade press, a project presence at exhibitions and conferences as well as regular newsletters and e-shots.

The FlowFree website has also been a useful dissemination tool. Throughout the duration of the project, the website has received up to 66,000 hits, with over 44,000 of those being received in the final 18 months of the project.

Conclusions

Objectives reached

The objectives of this project have been achieved and are summarised below:

- Prototype extruder systems were developed to investigate the use of super critical CO2 as a processing aid in polymer extrusion. The system was optimised to allow for full industrial, commercial development of the process.
- The process was installed at four different industrial sites and tested on an industrial scale with different materials and production requirements.
- The technical and commercial viability of the process at full industrial scale was demonstrated with the material to benefit most being highlighted.
- Dissemination and training activities were extensively carried out to allow for maximum exploitation of the technology.

Impact on industry

Patentable technology has been applied to the complete CO_2 assisted extrusion process in order to achieve maximum processing benefits. Arrangements are in place to actively seek exploitation within the extrusion sector of the plastics industry.

Trials with the industrial partners within the project have shown benefits which could potentially be implemented to improve their market competitiveness.

The modifications performed at each extrusion line worked well for the extrusion process assisted with the CO_2 injection.

The best results were obtained with PVC as it was easier to control foaming of the extrudate, and it was possible to obtain solid profiles with the same properties as those obtained with the traditional extrusion process.

Benefits in torque reduction, output increase and energy consumption reduction were measured and contrasted during the industrial trials. These benefits were more or less important depending on the polymer to be extruded.

The use of CO_2 did not affect the mechanical and thermal properties when compared with solid extrudates.