THE EFFECT OF PARTICLE SIZE TO SHORT TERM HYDROSTATIC INTERNAL PRESSURE PERFORMANCE OF CALCIUM CARBONATE FILLED POLYPROPYLENE BLOCK COPOLYMER COMPOSITE PIPES

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Abstract - In this study, short-term (ST) hydrostatic internal pressure (HSP) of micro particulate calcium carbonate (CC) filled polypropylene block copolymer (PPB) pipes were investigated according to the (CC) content. There are many studies demonstrate the CC particle size effect on the mechanical properties of Polypropylene (PP). To analyze these materials high pressurized methods are used like injection molding and hot pressing, similar to their regular production method. So the studies and information gathered on the effects of the filler amount are limited with the uniaxial test results. However, the PPB/CC composite pipe production by single screw extrusion method and the STHSP (short-term biaxial stress) effects on these products are still uncertain. Under these circumstances, the aim of this study is manufacturing CC filled PPB pipes to optimized Hoop stress properties. To investigate the CC filler content effect on composite PPB pipes, average particle size 1.65 μm, 2.15 μm and 5.00 μm CC filler content added to polymer 10% by volume. STHSP tests at 20°C water temperature were applied SDR (Standard Dimension Ratio) 11 and 75mm (D_o) diameter pipes according to ISO 1167 standard both for pure PPB and PPB/CC composite pipes. The experimental results bring out the fact that the CC particle size slightly affects the STHSP performance of the composite pipes. Moreover to that as the STHSP duration increase the damaged areas observed to be much smaller.

Keywords: Composite pipe, Hoop stress, Calcium carbonate, Particle size.

Introduction

The semi-crystalline thermoplastics and the applications that filled with various micro particles of these have been subjected to numerous studies. Many researches accomplished to understand the deformation characteristics of these thermoplastic materials [1]. Thermoplastic materials were manufactured by using CC [2] in different particle sizes with physical modification and weight percentage (wt %) amounts. To analyze the uniaxial mechanical properties and investigating the CC particle size [3] and content effect [4] to these thermoplastic materials, high pressurized methods are used like injection molding and hot pressing, similar to their regular manufacturing methods. However, the PPB/CC composite pipe production by single screw extrusion method and the STHSP (short-term biaxial stress) effects on these products are still uncertain. In this study regarding to ISO 6259-1, outer diameter (D) 75mm SDR (Standard Dimension Ratio) 11 pipes tested according to the standard ISO 1167 for hydrostatic pressure tests at 20°C water. The PPB/CC pipes were investigated according to the CC filler ratio. The effect of the particle size was referenced to pure PPB pipes in Hoop stress-time-temperature curve.

Experimental

Materials

Density of 900 kg/m^3 and mold flow index 0.30 g/min (230°C/2,16 kg) PPB material, which the commercial name is BorECO™ BEC5015 (Borealis A/S), is used. The mechanical properties tested from injection molded test specimens which 1500 MPa tensile modules, yield stress 30 MPa and 10% yield elongation are some instance information for mechanical properties given by the manufacturer product data sheet according to the standard ISO 527 (Borealis 22.01.2008 Ed.9). To investigate the filler content effect, 1.65 μm, 2.15 μm and 5.00 μm average particulate sized and density of 2700 kg/m^3 CC material, which the commercial names are in same order Mikrocarb-1, Mikrocarb-2X and Mikrocarb-5 (Mikro Mineral Endüstriyel Mineraller San. ve Tic. A.Ş.), were used. The white powdered CC material manufacturer information's were given in Table 1.

Table 1 Particle Size Distribution

Particle Size	Unit	Typical Value		
		CC1	CC2	CC5
Mean dia., d(0.5)	μm	1.65	2.15	5.00
Top Cut, d(0.97)	μm	7.50	11.60	28.00
Below 2 μm [min.]	%	57	48	30
Sieve 45 μm [max.]	%	0.01	0.01	0.1

Composite Preparation

The PPB/CC composite raw materials production was performed in screw length (L / D.) 38.5 and the helix length (L) 2700 mm twin-screw extruder (Coperion Werner and Plederer ZSK 70 Mcc). CC mineral filler added in to granule form PP-B pipe raw material at ratios of 10% by volume (25% by weight) and extruded with a twin screwed extruder machine, so mineral filled polyolefin compounds were obtained in granule form. The products were manufactured in a 500 kg/h extruder at -1 bars atmospheric pressure, between 220°C-245°C zone temperatures and 30°C set cooling water. Every batch was dried at 50°C for 4 hours.

Composite Pipe Extrusion

To manufacture the pipe according to the SDR (Standard dimension ratio) 11, outer dia. 75 mm pipes,

screw length (L/D) 36, screw dia. (D) 60 mm single screw extruder (Krauss-Maffei KME 1-60-36 B/R) was used. Pure pipes were manufactured at 200°C-260°C zone temperatures and 25°C cooling bath temperature. Besides pure pipe production, the 10% filled PPB/CC composite pipes were manufactured at 210°C-220°C zone temperatures and 25°C cooling bath temperature.

Hydrostatic Internal Pressure Test

Regarding to the test standard ISO 1167-2, from equation Eq.1 where the (D_O) outer diameter and l_2 is the testing clamp length, the sample length (l_1) was calculated 635 mm.

$$l_1 = 3.D_0 + 2.l_2 + 250mm \qquad \text{Eq.1}$$

To determine the smallest wall thickness (e) beyond the pipe length, a special fixture with 0,01mm precise measuring tool (Hi-Tech) is used. To determine the pipe sample's average outer diameter (d_e), the outer diameter (mm) where the thinner wall is the smallest and two sides diameters are 0,01mm precisely measured with a caliper (Mitutoyo).

$$P = 10 \cdot \sigma \cdot \frac{2 \cdot e}{d_e - e} \qquad \text{Eq.2}$$

The HSP tests were done at Blue Line 1586-0212 (Institut für Prüftechnik Geraetebau GmbH & Co.KG) testing machine in Kocaeli University. Regarding to ISO 1167-1 type-A end caps were used in tests. The internal pressure (bar) applied to the pipes are calculated from Eq.2 [Where (σ) indicates the Hoop stress (MPa), (d_e) average outer dia. of pipe (mm)] Pipes are stored at least 150 hours in well-conditioned and free of sunlight. The pipes are tested at 20°C±0,2°C temperature range, at calculated nominal pressure and can be diagnosis in circumstances like 1% pressure decrease and 2% pressure rise. To determine the HSP performance of the pipes at least 5 sample is used for each test.

Results and Discussion

Mainly we should note in here that pure PPB, PPB/CC composite materials, were used without being exposed to the effects of the parameters used in twin-screw exturision process. In this context, the pure PPB and PPB/CC composite pipes HSP properties were determined. To prepare the Hoop stress-time-temperature curves, for 20°C test temperature, regardless from the outer diameter and wall thickness of pipe, 16MPa Hoop stress value is taken as a reference from ISO 15494, at minimum required hydrostatic strength curve causes damage at 1 hour. This reference is chosen for a start value (16MPa = %100) to demonstrate the increases or decreases by percentage in graphics. The dimensions recorded on the SDR 11 and D75mm pure and PPB/CC composite pipes were shown in Table 2. The average dimensions plotted on the Table 2 were found to be less than Minimum tolerance values in ISO 15874. In Fig. 5, there is Hoop stress-time-temperature curves belongs to short-term HSP performance of SDR11, 75mm D filled with average particulate size 1.65 μm (CC1), 2.15 μm (CC2) and 5.00 μm (CC5) composite pipes.

Table 2 Average pipe section and wall thickness of pure PPB and PPB/CC composite pipes.

	Test hour	D_O, (mm) min	avr.	max	e, (mm) min	max
Pure PPB	≤ 10	74,59	74.97	75,39	6.83	7.28
	≤ 200	74.62	74.93	75.29	6.84	7.16
PPB / CC1	≤ 10	74.98	75.20	75.39	6.76	7.15
	≤ 200	74.95	75.25	75.58	6.73	6.88
PPB / CC2	≤ 10	75.02	75.19	75.43	6.88	7.13
	≤ 200	75.06	75.28	75.51	6.86	7.12
PPB / CC5	≤ 10	75.23	75.39	75.54	6.98	7.20
	≤ 200	75.16	75.34	75.60	6.91	7.11

When Fig. 5 analyzed, the damage occurred by Hoop Stress values of PPB/CC1, PPB/CC2 and PPB/CC5 composite pipes were decreased in the same order 22.4%, 19.7% and 21.3%. As a result it can be stated here that there is a slight effect of CC particle size on the STHSP performance of PPB/CC composite pipes.

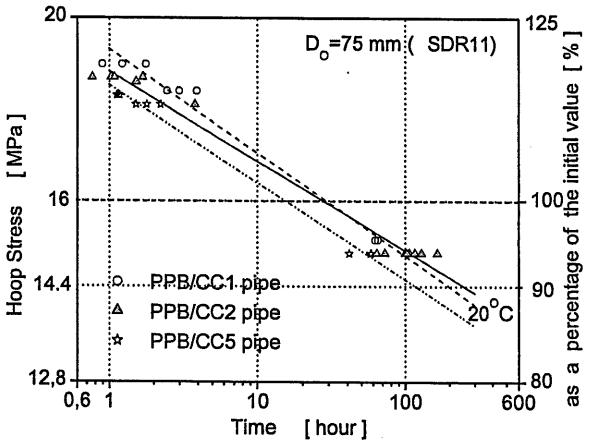


Figure 1 PPB/CC1, PPB/CC2 ve PPB/CC5 kompozit boruların çevresel gerilme – zaman – sıcaklık eğrileri

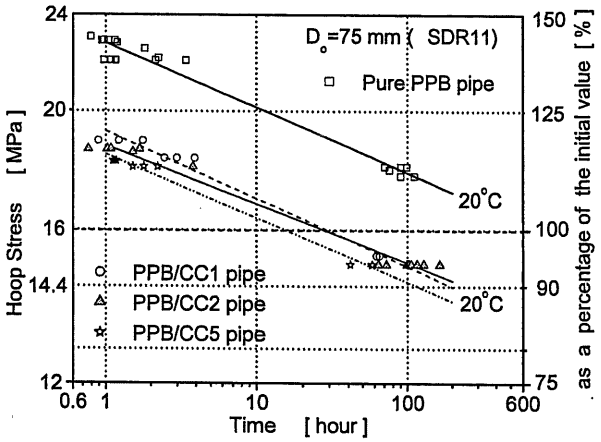


Figure 2 The Hoop stress-time-temperature curves of pure PPB, PPB/CC1, PPB/CC2 and PPB/CC5 komposite pipes.

The Hoop stress-time-temperature curves of pure PPB, PPB/CC1, PPB/CC2 and PPB/CC5 komposite pipes were given in Fig. 2. When Fig. 2 analyzed, first of all

it was observed that the 10% CC content has affected the HSP performance of pure PPB pipe. Then it was observed that as well as the 15.4% decrease effect of 10% CC content in PPB, CC particulate size showed approximately 4% decrease. In Fig. 3 according to ISO15494 minimum required hydrostatic strength time temperature curve for pure PPB, PPB/CC1, PPB/CC2 and PPB/CC5 composite pipes were given.

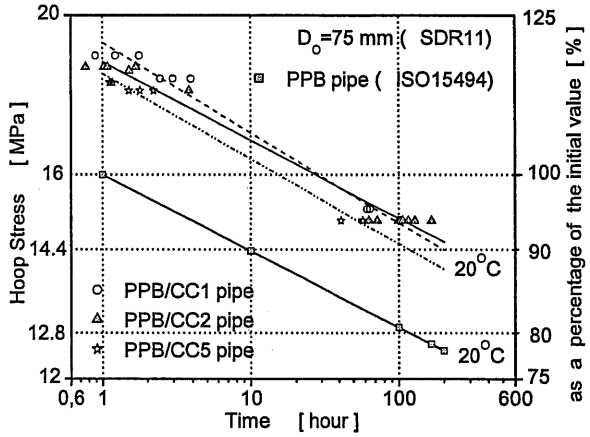
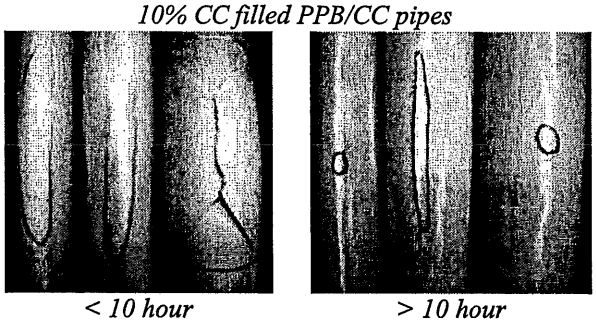


Figure 3 According to ISO15494 minimum required hydrostatic strength time temperature curve for pure PPB, PPB/CC1, PPB/CC2 and PPB/CC5 composite pipes

When Fig. 3 analyzed, it is observed that the Hoop stress values for PPB, PPB/CC1, and PPB/CC2 and PPB/CC5 composite pipes were higher than the minimum required hydrostatic strength time temperature curve for pure PPB (acc. to ISO 15494). Then the decrease of stress value in PPB/CC1 PPB/CC2, PPB/CC5 composite pipes, between the same durations, in the same order of about 22.4%, 19.7% and 21.3%, for the standard associated with PPB pipes, as reference standards predict value 19.4% the amount of change in performance were also detected higher.

10% CC filled PPB/CC pipes



< 10 hour *> 10 hour*

Pure PPB pipe

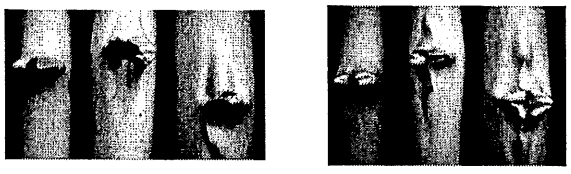


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Figure 4 Damages on the pipes under STHSP.

In Fig. 4 the damages on pipes were shown under 1 to 100 hours HSP (biaxial stress) effect. It was observed ductile fracture type by the decreasing HSB duration in pure PPB pipe. Against to pure PPB, it was observed brittle fracture on the composite pipes. It's been figured out that there wasn't any change in the pure PPB pipe, besides the composite pipe showed a ductile-brittle mixed damage type by the duration extends.

Conclusions

The experimental results bring out the fact that the CC particle size slightly affects the STHSP performance of the PPB/CC1, PPB/CC2 and PPB/CC5 composite pipes. However, against the 10% CC filler content effect in pure PPB pipe, the effect of the CC particle size (4%) considered to be negligible. The Hoop stress values of the composite pipes under HSP determined to be higher likewise the uniaxial tensile test by the decrease of particle size. But it was observed that the particle size has no effect on the Hoop stress by the increasing duration under HSP. Besides, the CC filled in different average particulate sized (1.65 μm, 2.15 μm ve 5.00 μm) SDR11 D_O=75mm composite pipe failures (crack sizes) were observed to be minimized by the increasing duration of HSP, on the other hand the CC particle size effect was considered to be negligible. For future studies it is recommended to use hydrophobic featured CC materials and lower filler contents.

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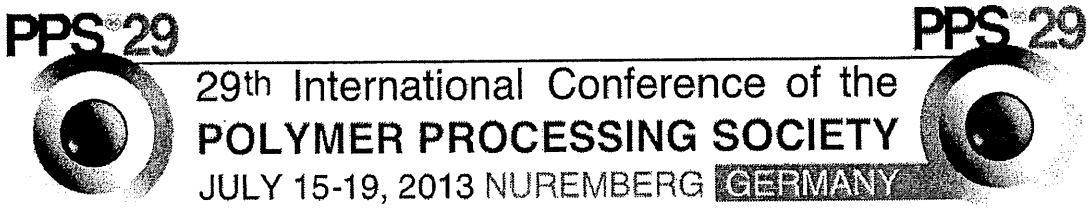

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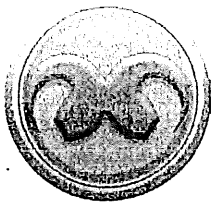

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Membership for the PPS is open to all researchers in the field, and to all persons who feel the activities of the society advance their professional development.

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8th	New Delhi, India, 1992
9th	Manchester, UK, 1993
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12th	Sorrento, Italy, May 27-31, 1996
13th	New York City, NY, USA, 1997
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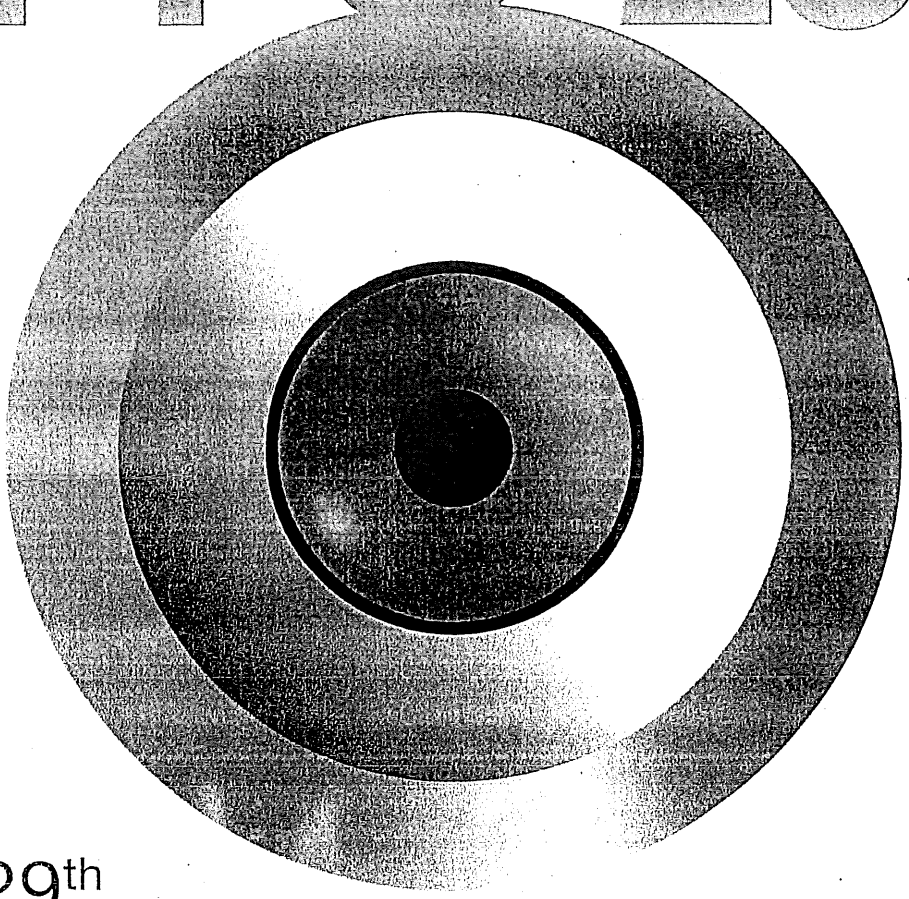

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