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Assessment of Effects of Heat-Setting Temperatures on Tensile Properties in Polypropylene Single-Fibres

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Abstract
This study on effects of heat-setting temperatures on tensile properties of Polypropylene (PP) single-fibres was initiated by a non-woven manufacturing company. The study is therefore important as it is addresses the real need of the industry to have more comprehension on effects of heat-setting, in order to develop a better end-products. The research was conducted on non-conventional single-fibre tester Favimat-Robot (Technico), Germany at the Textile Physical testing Laboratory of Vakgroep Textielkunde Universiteit Gent (Ghent University), Belgium. Testing of PP single-fibres was limited to the following parameters: Elongation (Fmax), Maximum force, Work (break), Tenacity, Linear density, Time to rupture and Modulus. The collected data was statistically analyzed by Analysis of Variance ANOVA test, using STATGRAPHICS Centurion XVI.II software, while chart was generated using Microsoft Excel program. Major findings of this study reviled that all tensile test-parameters show a high variation of different extent. Elongation and Time to rupture parameters showed increase with increasing temperature, while none of the heat-setting temperatures had a clear effect on the rest of the test-parameters. ANOVA analysis and Multiple Range Test denoted a statistically significant difference for Tenacity, for Linear density, and for Time to rupture. The study recommends further research-experiments, where the temperature-range should be broken into smaller segments by increasing subject-temperatures from two to five (adding 125,130 and 135^{o} C).
Key words: Favimat-Robot, tensile properties, Polypropylene, single-fibre.

1. Introduction

1.1. Polypropylene fibre

Polypropylene (PP) fibers are new generation chemical fibers, and PP is the first stereo-regular polymer to have achieved industrial importance. The fibres from PP were introduced to the textile arena in the 1970s and have become an important member of the rapidly growing family of man-made/synthetic fibres. Today PP enjoys fourth spot behind the "big three" fibre classes, i.e. polyester, nylon and acrylic. About 4 million tones of PP fibers are produced annually worldwide (Buchanan, 1981; McIntyre ed., 2005).

PP is the principal fibre of commercial importance of "olefin" or 'polyolefin" family, in which the synthetic polymer is composed of at least 85% by mass of polyolefin units (McIntyre& Daniels, 1995; Buchanan, 1981). PP can be made from the monomer propylene by Ziegler-Natta polymerization and by metallocene catalysis polymerization (McIntyre& Daniels, 1995).

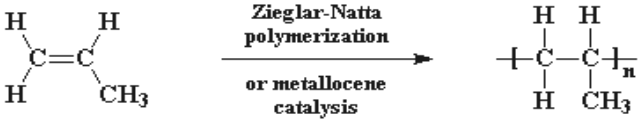

Figure1: PP polymerization ((McIntyre& Daniels, 1995).

PP fibre is uncomplicated to process, is a good substitute for numerous other materials; is quite readily recycled and is considered to have little adverse environmental impacts. PP fibres are to be found, for example, in carpets and other household textiles, motor vehicles, geo-textiles, healthcare and hygiene products, filters, sacks and bags, cables, ropes, netting, agricultural and horticultural products (McIntyre ed., 2005).

1.2. Selected PP Properties

PP selected properties can be summarized as follows (McIntyre ed., 2005):

1. PP is a light fibre, its density (.91 gm/cm^3) is the lowest of all synthetic fibres.
2. It does not absorb moisture. This means the wet and dry properties of the fibre are identical. Low moisture regain is not considered a disadvantage because it helps in quick transport of moisture as is required in special applications like babies' ever-dry nappies.

3. It has excellent chemical resistance. PP fibres are very resistant to most acids and alkalis.
4. The thermal conductivity of PP fibre is lower than that of other fibres and may be used in applications as thermal wear.
5. PP fibres possess an excellent strength (depends on degree of polymerization).

PP fibers have a softening point in the region of 140°C and a melting point at (160-170°C). PP has the lowest softening point of all synthetic materials. Nevertheless, this temperature is sufficiently high to enable the fibre to be processed satisfactorily in almost all normal manufacturing processes. On the other hand, the high melting point of PP is an advantage in many nonwovens processing steps. The maximum processing temperature of PP fibre is approximately 140°C. Prolonged exposure to elevated temperatures will cause degradation of the fibre, but anti-oxidants are incorporated in PP fibres to protect them during processing and at normal service temperatures. PP fiber can be softened sufficiently to bond to one another without destroying fiber properties. Nonwoven fibers made from PP can therefore be fusion-bonded, eliminating the need for chemical binders. The benefits of this technique include both energy saving and environmentally friendliness (McIntyre ed., 2005).

1.3. Importance of PP

In the production of PP fibres, continuous filaments are first formed by melt spinning, and these filaments are then drawn. The structure and properties of the final production are determined by the details of both production steps (spinning, drawing and heat-setting) and the properties of the resin used (Ahmed, 1982; Samuels, 1974; Lu&Spruiell, 1974).

Polypropylene is extremely versatile as a fiber-forming material. Since its introduction into the textile industry in the 1970s, the list of successful products and markets for PP fiber has increased exponentially (Richard, 1999). Because of its superior performance characteristics and comparatively low-cost, PP fiber finds extensive use in the nonwovens industry.

The extensive commercial interest in melt-spun polyolefin fibres, and especially PP fibres, stems from their mechanical properties. As with other synthetic fibres, there is particular emphasis in this regard on fibre strength, as indicated by tenacity, and fibre stiffness, as indicated normally by initial modulus (Elongation to break) is also an important technological property (Tomka, 1996).

1.4. Tensile properties of textile fibres

Performances of fibers under different forces and deformations, which are applied along its longitudinal axis, are defined as the *tensile properties* of fibers.

A fiber's tensile properties are important because it must possess enough strength to with-stand processing by the machinery and also provide the desired durability for the non-woven fabric produced (Wulfhort, 2006). It also must possess some elasticity so that it does not break during processing. If the fiber is too elastic it will cause processing problems as well. The tensile strength of a fiber is determined by tension tests that apply a tension load to a fiber until the fiber breaks. The load when the fiber breaks is the breaking load. This is in turn used to express the tensile strength of the fiber by reporting the force per unit of linear density. Therefore, the tenacity of a fiber is expressed in grams per denier (Hudson et. al., 2005). The tenacity is not only important for determining the physical strength properties of the non-woven fabric being produced, but it is also important for withstanding fiber breaking-tensions during processing (Hsu, 2011).

1.5. Problem statement and research justification

Finding new products, alternative raw materials, and processing technologies more "environmentally friendly", obtained with new processes and conditions, is central for further developments of textile industry (Bunsell ed., 2009).

Given the importance of understanding mechanical behavior in textile fibres, it is reasonable to assume that mechanical behavior of these textile materials has already been reported since long time in literature (Bunsell ed. 2009; Abbott et. al., 2009). Despite these efforts, very little is available in the open literature about the tensile behavior of single individual fibers, which is important from the perspective of a textile materials engineer, designing superior end-products for textile industry.

The tensile strength of the fibers after treatment is of utmost importance to establishing the efficacy of a heat-setting procedure. Single-fiber tensile testing is used to evaluate the strength of fibers after the heat-setting treatment. While extensive research has been done on the tensile properties of single-fibers (Bunsell, ed., 2009; Liu, 2005; Hsieh et al., 2000; Foulk & McAlister, 2002), little information (if at all) is available on the relationship between heat-setting temperatures and tensile properties.

With respect to the above, the main objective of the present study is to attempt to characterize the thermo-tensile behavior of PP fibres, with the aim of manufacturing non-woven materials with high mechanical performance. The study is important because it addresses the real need of non-woven industry in their endeavor to produce superior end-products; the study will also contribute to the body of knowledge on single PP fibres tensile behavior.

2. Materials and Methods

2.1. Parameters tested

Tensile testing of single fibres represents one of the most important quality control testing methods in chemical-fibre production. In addition to the (linear density-related) breaking force and breaking elongation, other parameters such as modulus, intermediate values of the force/elongation curve, e.g. force values at specified elongations, work to rupture or characteristic values for elastic- and plastic deformations, can be obtained. The fiber strength properties are derived from the peak load of each fiber. When the fiber is mounted between the two clamps of the machine, it is loaded until it breaks.

2.1.1. Linear density (dtex)

The FAVIMAT automatically determines the linear density of single-fibres using the vibration method, according to ASTM D 1577. With this testing method the resonance frequency of the sample is measured at constant gauge length and known pre-tension; the data obtained is then used for calculating the linear density according to the following formula (Doguc, 2002).

$$T_t = F_V / (4 \times f^2 \times L^2)$$

T_t = linear density
F_V = pre-tensioning force
f = resonance frequency
L = test section length

2.1. 2. Tenacity (cN/dtex)

Tenacity or specific stress is the customary measure of strength of a fiber, which is applied along the longitudinal fiber axis, is measured in *N/tex*. Since the regular SI unit for specific stress is Nm/kg, they use Newton per tex (*N/tex*) and *centy* or *milli*-Newton per tex (cN/tex or *mN/tex*) for lower stress.

2.1.3. Elongation (%)

Fiber breakage can be expressed by the percentage of elongation too. In other words, the breaking elongation gives a measure of the resistance of the material to elongate and, finally, break. It means how much the fiber will extend or elongate for finally the break to occur. It is an essential and critical property when a fiber or an assembly of fibers is subjected to stretching. Unlike the strength which can be stated in different units, elongation to break is simply normalized as fractional strain or percentage extension.

2.1.4. Maximum Force (cN) - this is a maximum force that a fibre can withstand without being ruptured.

2.1.5. Work (break) /work to rupture (cNcm): the area contained by the force/elongation curve up to the point where the breaking force is reached. This is a measure of the toughness of a fibre.

2.1.6. Time to rupture- time in seconds taken a fibre to rupture

2.1.7. Modulus of Elasticity (cN/dtex)-ratio of pressure (stress) applied to a fibre to the resistance (strain) produced by a fibre. It is the ratio of longitudinal stress to strain, it is also called Young modulus.

2.2. Subject- Samples

PP fibres were being produced by the same manufacturing company, on the matching equipment, under the similar processing conditions, of the same linear density and within the limited time-frame. An oven with controlled heating-rate was used (the temperature inside the oven was monitored using hp 3497 Data acquisition system).

Three sets of representative samples of PP single-fibres were prepared in accordance with BS 2545(subject-sample #1- reference; sample#2- set at 120^{o}C, and sample#3- set at 140 ^{o}C).

Fifty fibres (on average) from each of the subject-samples were tested according to the Favimat-Robot Standard Test procedure (Textechno, 1999). In particular, for sample #1- 50 single-fibres were tested, while for sample# 2- 51 tests were conducted, out of which 2 tests reported as not possible. For sample# 3- 52 tests were done (2 tests were reported as not possible).

2.3. Equipment and settings used.

Testing of PP single-fibres was conducted at Textile Physical testing Laboratory of Vakgroep Textielkunde Universiteit Gent (Ghent University), Belgium. The Favimat-Robot single-fibre tester was used in this study; it is semi-automatic, microprocessor-controlled testing equipment, working according to the principle of constant rate of extension (DIN 51221, DIN 53816, and ISO 5079). It allows the force to be measured at a high resolution of 0.1 mg. Moreover, this instrument is equipped with an integrated measuring unit for linear density (in dtex). The linear density is measured according to the vibroscopic method (ASTM D 1577, BISFA 1985/1989 chapter F) using a built-in automatic measuring head. The fiber is loaded to a predetermined specific tension at a predefined speed, and then exited with an electro-acoustic sinusoidal vibration. The resonance frequency is detected with an optoelectronic sensor. For simplicity of the calculation, uniform mass distribution and circular cross-section of the fiber is assumed, and bending rigidity is disregarded.

Fiber strength values obtained from the FAVIMAT tester are simply the peak load of the fiber. For this test, the fiber, which is mounted between the two clamps of the tester, is loaded until it breaks.
For comprehensive procedure of testing and conditioning used, on Favimat, refer to Starovoytova et.al. (2015). Pre-calibration was done according to Textexhno (1999).
Settings used: Load cell 210 cN, Gauge length 20mm, and Nominal L.D. 16.00 dtex. The tensile properties were tested with a gauge length of 20 mm, a test speed of 20 mm/min, and a pretension of 0.5 cN/dtex. For the linear density, a test speed of 20 mm/min and a pretension of 0.8 cN/dtex were applied.

2.4. Data analysis' tools
The collected data was analyzed by the Analysis of Variance ANOVA test, using STATGRAPHICS Centurion XVI.II software, while charts were generated using Microsoft Excel program.

3. Results

The test-results from Favimat-Robot are presented in Table 1, Table 2 and Table 3, for Sample#1, Sample#2, and Sample#3 respectively. The mean value ("central tendency") for each of the tested parameters is presented in Figure2 and Figure3.

Table1: Test-results for sample#1

Statistics	-N-	-X-	-S-	-CV-	-Q(95%)-	-MIN-	-MAX-
Elongation(Fmax)	50	139,11%	41,80	30,05	11,87	4,94	222,40
Maximum force	50	49,87cN	7,79	15,61	2,21	15,36	61,36
Work(break)	50	118,59cN*cm	41,90	35,33	11,90	0,97	201,48
Tenacity	50	2,75cN/dtex	0,36	13,22	0,10	0,80	3,45
Linear density	50	18,30dtex	2,66	14,55	0,76	11,49	23,78
Time to rupture	50	85,35sec	24,42	28,61	6,94	3,22	136,01
Mod.E1(0..1%)	50	21,39cN/dtex	4,53	21,16	1,29	11,92	32,72
Mod.E2(2..3%))	50	13,11cN/dtex	1,44	10,97	0,41	10,44	16,80

Table2: Test-results for sample#2

Statistics	-N-	-X-	-S-	-CV-	-Q(95%)-	-MIN-	-MAX-
Elongation(Fmax)	49	143,20%	42,42	29,62	12,18	29,45	249,89
Maximum force	49	49,22cN	3,97	8,06	1,14	35,01	55,49
Work(break)	49	117,85cN*cm	35,70	30,29	10,25	17,44	192,20
Tenacity	49	2,99cN/dtex	0,27	8,99	0,08	2,13	3,70
Linear density	49	16,54dtex	1,62	9,81	0,47	13,76	19,45
Time to rupture	49	87,53sec	25,79	29,46	7,40	18,77	150,78
Mod.E1(0..1%)	49	24,08cN/dtex	4,70	19,52	1,35	11,58	33,39
Mod.E2(2..3%))	49	13,40cN/dtex	2,42	18,04	0,69	7,44	20,11

Table3: Test-results for sample#3

Statistics	-N-	-X-	-S-	-CV-	-Q(95%)-	-MIN-	-MAX-
Elongation(Fmax)	50	159,37%	53,87	33,80	15,30	40,22	308,24
Maximum force	50	48,16cN	3,88	8,06	1,10	38,14	55,00
Work(break)	50	126,55cN*cm	40,97	32,37	11,64	25,08	213,82
Tenacity	50	2,91cN/dtex	0,31	10,63	0,09	1,97	3,35
Linear density	50	16,70dtex	1,94	11,59	0,55	13,33	22,66
Time to rupture	50	97,95sec	30,08	30,71	8,54	36,24	186,26
Mod.E1(0..1%)	50	21,93cN/dtex	4,56	20,77	1,29	10,52	30,29
Mod.E2(2..3%))	50	12,59cN/dtex	2,53	20,09	0,72	6,41	17,94

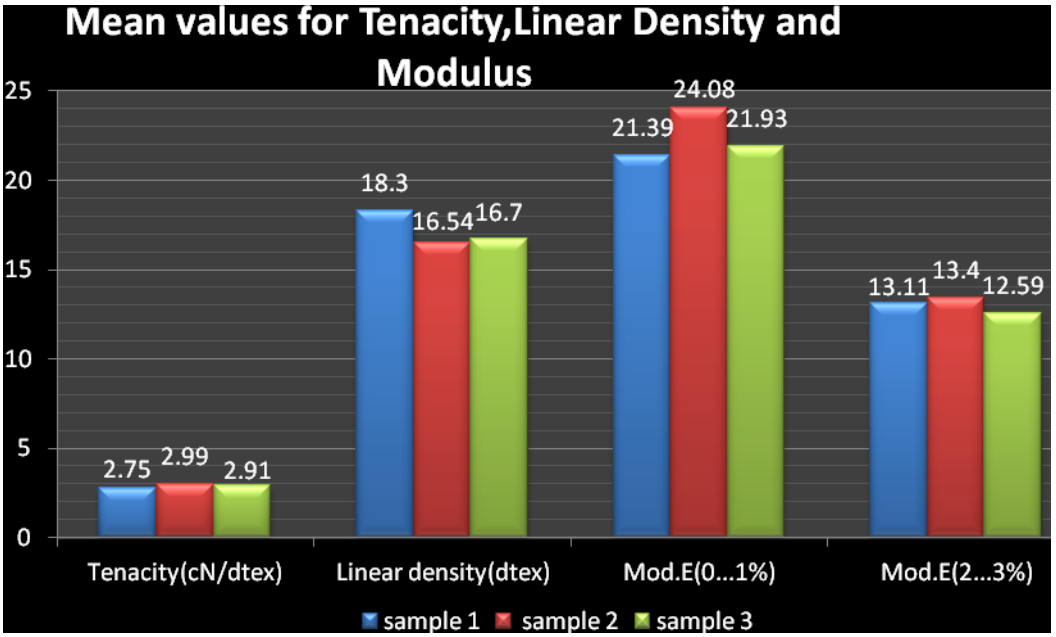

Figure2: Mean value for Tenacity, Linear Density and Modulus.

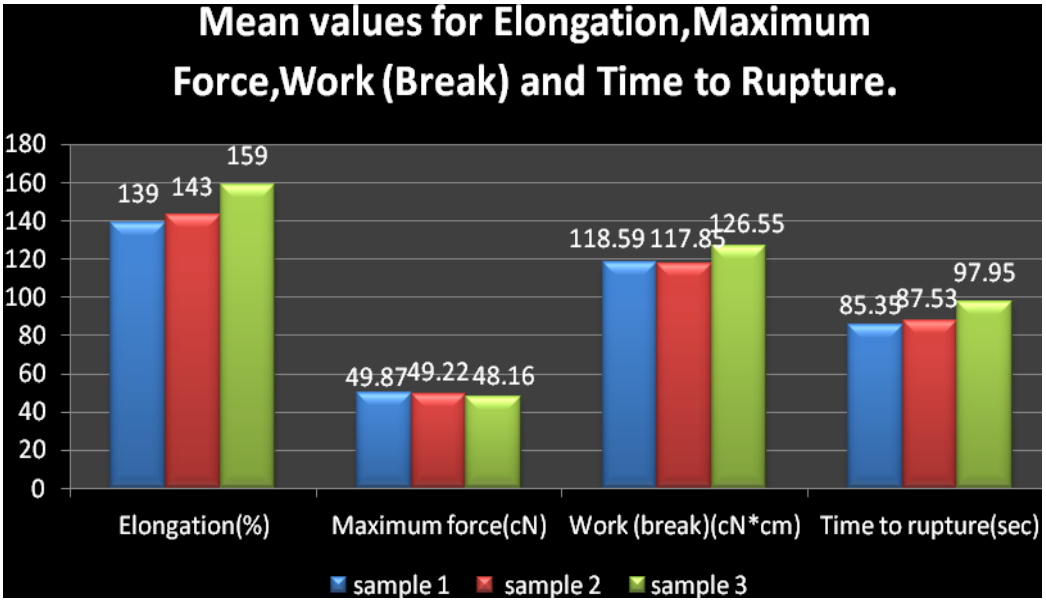

Figure3: Mean value for Elongation, Maximum Force, Work (break) and Time to rupture.

4. Data analysis

The results of ANOVA analysis are presented in Table 4.

Table4: ANOVA Analysis of Variance test-results

Parameters	F-Ratio	P- value
Elongation	2.66	0.0731
Maximum force	1.21	0.3016
Work (break)	0.56	0.5698
Tenacity	7.60	**0.0007**
Linear density	11.35	**0.0000**
Mod. E (0...1%)	5.33	0.0058
Mod.E2 (2…3%)	1.72	0.1828
Time to Rupture	3.13	**0.0465**

5. Discussions

The first observation that should be made is that all test-parameters show a very high variation. For Work (break) it is the highest, ranging CV from 30.29-35.33; the next highest CV is for Elongation (29.62-33.8); followed by Time to rupture (28.61-30.71). Although the rest of the test-parameters show comparatively lower CV, it is still rather high variation, as the lowest CV is for Tenacity ranging from 8.99 to 13.22. The high variability (intra-individual) is confirmed by other authors (Moore, 2012; Huabing, 2003; Van Nimmen et. al., 2005; Liu, 2005; Schultz, 2001).

From Figure 2 and Figure 3 it is seen that only Elongation and Time to rupture showed increase with increasing temperature. From the results it can be also concluded that none of the heat-setting temperatures had a clear effect on the rest of test-parameters, such as: Linear Density, Tenacity, Modulus, Work (break), and Maximum Force.

With regard to ANOVA analysis (Table 4), since the P-value of the F-test is less than 0.05, there is a statistically significant difference between the means of the 3 variables at the 95.0% confidence level (highlighted in red). To determine which means are significantly different from which others, Multiple Range Tests was done. The method currently being used to discriminate among the means (Multiple Range Test) is Fisher's least significant difference (LSD) procedure. With this method, there is a 5.0% risk of calling each pair of means significantly different when the actual difference equals 0. The test denoted a statistically significant difference for Tenacity-sample1 and sample 2; for Linear density- sample1 and sample 2; and for Time to rupture- sample 3.

This concise study was restricted to just two subject-temperatures (120^{o}C and 140^{o}C) and in addition, it is manifested high variability of test- results, accordingly, the study was incapable to achieve the required level of conclusiveness, and consequently, to categorically identify the optimum heat-setting temperature and to ascertain any influences of heat-treatment temperatures on crimp parameters of PP single-fibres. The study, therefore, recommends, in further research-experiments, to break the temperature-range into smaller segments by increasing subject-temperature from two to five, with inclusion of 125,130 and 135^{o} C.

6. Conclusions and Recommendation

The influence of heat-treatment temperatures on tensile properties in PP single-fibres has been studied through the high-resolution and-sensitivity testing equipment FAVIMAT-Robot by Textechno. It was observed that all tensile test-parameters show a high variation of different extent. Elongation and Time to rupture parameters showed increase with increasing temperature. From the results it can be also concluded that none of the heat-setting temperatures had a clear effect on the rest of test-parameters, such as: Linear Density, Tenacity, Modulus, Work (break), and Maximum Force. ANOVA test and Multiple Range Test denoted a statistically significant difference for Tenacity, for Linear density, and for Time to rupture.

The study recommends, in further research-experiments, to break the temperature-range into smaller segments by increasing subject-temperatures from two to five, by adding 125,130 and 135^{o} C.

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