Comparison of mechanical properties of different particle sizes of recycled polycarbonate at higher temperature

Vojtech Senkerik, Michal Stanek, Miroslav Manas, David Manas, Adam Skrobak, and Jan Navratil

***Abstract*—**This research paper deals with behavior of particles of a recycled material at a higher temperature depending on the particle size. Behavior is tested by mechanical properties. During grinding particles are formed which have a different size, shape and surface, from larger pieces to dust particles. During processing these particles melt at different rates depending on their size. For example they can cause material degradation or lack of melt homogeneity. Several recycled mixtures were prepared that had differed particle size after crushing. The recycled material is always the same as is the original material. The studied material was high-heat polycarbonate. Testing was performed using a tensile test, Charpy impact test and hardness test. Specimens were prepared by the mostly used technology for production products, which is injection molding. Each mixture is one by one loaded by high temperature 100°C and consequently tested. This temperature was chosen because we encounter products made with recycled material additive, which can be used at elevated temperatures. When comparing a virgin polycarbonate with recycled mixtures, the particle size of the recycled material affects some of the material parameters quite substantially, but it had no effect on some other properties.

***Keywords*—**recycled material, particle size, sieving, preparation of recycled material, temperature, polycarbonate

I. Introduction

PLASTICS recycling continues to grow around the world. Mechanical recycling is the most common method of recycling. Here plastics are physically ground back to a suitable size (regrind) and reprocessed. The end use can be the original one or something different.

In the plastics industry it has long been common practice to reprocess waste material arising from normal production. This in-house recycling, known as primary recycling, makes economic sense as it reduces both production waste and utilization of raw materials. For example, with injection molding, regrind from start-up waste and production waste such as reject parts, can be fed directly back into the production machine.

Within a closed loop cycle, it is easy to recycle materials and this is the reason that primary recycling is so commonplace. The key is having the knowledge of, and confidence in, the materials that are being used.

Injection molding has in last several decades become very popular technology in plastic manufacturing. It allows quick and precise manufacture of different polymer based products not only for daily life, but also for specialized uses.

Defective injects, waste and runner system originating during injection molding can be processed several times. This way of processing is frequently used because of high portion of the waste, especially while manufacturing small injects. For this reason is unpolluted waste cut and crushed. Material modified by this process is once again granulated and mixed with pure granulate and consequently utilized. Using this mixed material has usually no significant influence on physical-mechanical properties or surface appearance. Level of properties degradation depends on levels of crushed material in original material. Transparent and highly stressed materials cannot be mixed, because of high demands. This article focus to this method type of recycling - mechanical recycling.

Injected product can be made even with fifteen to thirty percent amount of waste in origin material without significant influence on its properties. Material properties gradually decrease with higher percents of waste in mix. In some cases (low-end simple injects) parts can be made even from hundred percent of waste.

Although recycling has a very long history, it is only relatively recently that environmental protection and waste management issues have come to the forefront of both public and political awareness. The removal of plastics from both the waste streams and from landfill have since become areas of major interest.

Outside the fields of expertise, generally little is known about either plastics or their recyclability. A number of specialist books are available; however none has addressed the

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need to cover the pertinent principles at an introductory level.

For reclaiming used material or recyclate outside of this scenario, the situation is slightly different and greater effort scenario, the situation is slightly different and greater effort may be required on the part of the reprocessor.

The material from external sources may be received in a variety of forms such as bales, mouldings or large lumps. It will probably need to be reduced in size, cleaned, separated and possibly recompounded and regranulated before it can be reprocessed in production. Often little is known about the history of the material to be recycled.

The characteristics of plastics can change depending on the exposure to thermal, mechanical (shear), oxidative and photochemical degradation processes. The characteristics of the recyclate may be quite different from those of the original virgin plastic.

Ideally, to produce high quality products, high quality materials are required. For this, consideration must be given to a number of factors. It must be determined as to whether the material is pure or commingled and whether it is contaminated, for example, with metal or wood. For ease of feeding into the processing machines be they injection moulding, extrusion or blow moulding, the size and shape of the regrind (that is the bulk density) must be suitable. If the material is hygroscopic (water absorbing), for example polyamide, it may require pre-drying. Finally, should the recyclate be reprocessed on its own, mixed with other virgin material or modified with additives?

Within a closed loop cycle, it is easy to recycle materials and this is the reason that primary recycling is so commonplace. The key is having the knowledge of, and confidence in, the materials that are being used.

One example of a closed loop cycle in action is seen in the automotive industry. Since Volkswagen have recycled scrap bumpers made of a modified grade of polypropylene (PP). Their supplier reclaims the material, which is then mixed with virgin and returned to the bumper production process. The properties of the bumpers produced are as good as those made using virgin material alone. In tests it was found that no significant difference in characteristic properties occurs until the material has been melted and extruded eight times.

The effects on material properties of mechanical recycling can be explored by repeated cycling of material through processing machines. Material from each cycling loop can be assessed. For example, in the production of the Volkswagen bumpers mentioned earlier, tests found that properties changed significantly, only after eight cycles of reprocessing. Experiments of this kind have shown that short-term properties do not vary too greatly if the material does not contain glass fibres. Glass fibre is a very common reinforcement used in plastics.

However, this material tends to become damaged when reprocessed. The mechanical strength of the plastic is dependent on the length of the fibres used and the act of processing reduces this residual length. However, a word of caution should be applied here. Note that these are short-term properties only. The long-term effects of repeated processing on plastic properties are still under investigation. Whether these materials, when mixed with virgin, will undergo accelerated degradation is still the subject of current research. It does seem clear however, that incorrect processing parameters (too high temperatures) cause far more damage to the plastics than repeated processing at suitable temperatures. [1 - 4]

II. Experiment

The aim of this research paper is to study the effect of particle size of recycled polymeric material on mechanical tensile properties at increased temperatures. The goal is to perform an experiment in which the products of the studied polymer will be crushed to crushed material (recycled material) and then reprocessed into new products. These are then subjected to mechanical testing. Specimens were prepared by the most common technology for production of plastic products, which is injection molding.

A. Material

Tested polymer was polycarbonate Makrolon 2205. This material is formed by a condensation polymerization resulting in a carbon that is bonded to three oxygen atoms. The most common system for this polymerization is formed by a reaction of bisphenol A and phosgene. Applications of polycarbonate are almost always those which take advantage of its uniquely high impact strength and its exceptional clarity. These unique properties have resulted in applications such as bulletproof windows, break resistant lenses, compact discs, etc. Recently, more interest has risen because of the low flammability of polycarbonate.

Polycarbonate has a glass transition temperature of about 147 °C, so it softens gradually above this point and flows above about 155 °C. Tools must be held at high temperatures, generally above 80 °C to make strain- and stress-free products. Low molecular mass grades are easier to mold than higher grades, but their strength is lower as a result. The toughest grades have the highest molecular mass, but are much more difficult to process. [5]

B. The specimen preparation

The first task was to produce products by injection molding technologies. These parts were made using recycled material. Injection was carried out on injection molding machine made by Arburg. Specifically, it was an injection molding machine Arburg 470 C. Each of the specimens were left to condition for 24 h before testing by the following method.

Table I Process parameters

Melt temperature	300 °C
Mold temperature	100 °C
Ejection temperature	130 °C
Injection speed	60 mm.s^{-1}
Injection pressure	80 MPa
Holding pressure pressure	35 MPa
Holding pressure time	30 s
Cycle time	55 s

C. Sieving

Grinding of material was immediately followed by sieving. The purpose of the sieving was to eliminate the above mentioned very fine particles and dust. Sieving was carried out on laboratory sieve shakers AS 200 Basic figure.

Sieve mesh of size 2 mm was used for sieving, which caught the particles larger than 2 mm. This material is further called sieved crushed material. The size of mesh was chosen to achieve the size of recycled material to be as close as possible to the original material. The bowl was placed to the bottom to capture smaller particles than 2 mm and dust. Non-sieved crushed material then contain particles of all sizes and is very diverse.

After the crushed was made, preparation for the injection of new specimens made from this material began. Several recycled materials were made, all from original material. Samples with different percentage amount and a type of recycled material were subsequently tested.

For injection molding the following mixtures of these materials were used:

- virgin polycarbonate,
- sieved crushed material,
- non-sieved crushed material.

D. Composition of tested mixtures

Different compositions were prepared for the measurements of tested mixtures with differing sizes of sieved particles.

• Virgin PC - material directly from the manufacturer which has not yet been processed in any way, it is taken as a referential material

• Sieved crushed material – recycled crushed material containing similarly sized particles as the granules of virgin material, from this mixture small crushed particles smaller than 2 mm are removed

Fig. 1 Sieved crushed material (particles size larger than 2mm)

• Non-sieved crushed material - this recycled material is collected directly from the knife mill, contains particles of all sizes, from large particles to dust particles and therefore is heterogeneous.

• Crushed material 1 – 2 mm - this recycled material is sieved and has a particle size from 1 mm to 2 mm

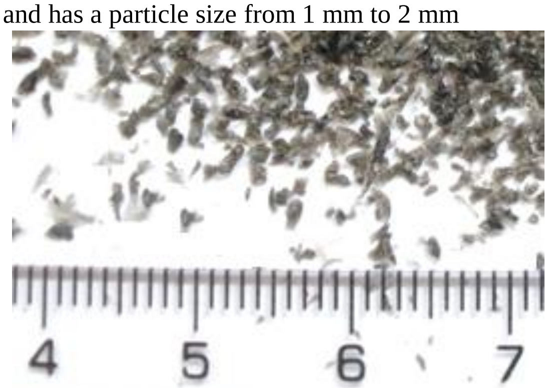

Fig. 2 Crushed material 1 – 2 mm

• Crushed material < 1 mm - this recycled material is sieved and has a particle size up to 1 mm, containing very small to dust particles.

Table II Tested mixtures

Composition of tested mixtures	Particle size [mm]
Virgin polycarbonate	granule
Sieved crushed material	$2 < x < 4$
Non-sieved crushed material	$0 < x < 4$
Crushed material 1-2mm	$1 < x < 2$
Crushed material < 1mm	$x < 1$

E. Testing

1) Tensile test

Tensile test was performed on a universal tensile testing machine W91255 from Zwick / Roel. The test was carried out according to EN ISO 527 standard. For each mixture 10 measurements were performed, which were then statistically evaluated.

Tensile testing can be thought of as a stretching test. Tensile properties are a method used widely to analyse the short-term stress-strain response of a material. Computer controlled tensile test machines are commonly employed, allowing one simple test to give a variety of information regarding strength, elongation and toughness. For this, a dumbbell-shaped test specimen is required such as the samples shown in Fig. 3.

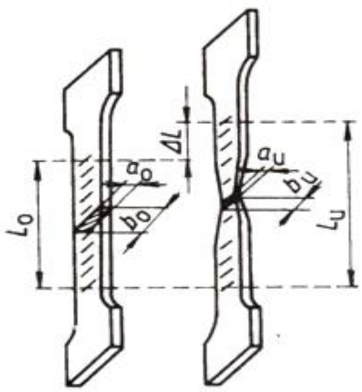

Fig. 3 Zwick/Roel type 1456

Standard procedures are used such as the International Organization for Standardisation method ISO 527 or the American Society for Testing and Materials method, ASTM D638 to ensure comparison can be made between materials. The shape and gradient of the curve produced describes the way the sample stretches or breaks, its strength and rigidity, and whether a material is brittle or ductile (pliant). [4]

2) Hardness test

Shore D hardness test was performed on the hardness tester from the OMAG company with type marking ART 13. The test surface of specimens must be cleaned before test. Testing was performed according to EN ISO 868. First, the sample was placed in a rack of hardness tester and then indenter started pressing to the surface of the test specimen. After the time interval of 5 seconds display shows the value of hardness Shore D. Hardness Shore D is determined by the depth of penetration of the indenter and the size of the scale is then 0-100 Shore.

3) Charpy impact test

The last mechanical test in order was notch toughness test alias Charpy impact test. Testing was performed on the Resil Impact Junior testing machine from company CEAST. The tests were carried out according to ISO 179. Preparation of individual samples proceeded before each testing. It consisted of making of notch in specimens. Notch was shaped into V-shape with depth of 2 mm.

Impact tests measure the ability of a material to withstand a high velocity impact, for example, as might be experienced by a plastic kettle dropped from a kitchen worktop. Again, standard methods (e.g., ASTM D256) and specimens are employed; the results give a measure of the toughness of materials. Generally, the methods used fall into two categories. In one, a pendulum strikes a sample and the energy required to break the test piece in one pendulum swing is noted. This is most often seen on data sheets as Charpy or Izod impact tests. Both Charpy and Izod use a standard striking energy.

Samples can also be notched, the size and shape of the notch is also standardised. This ensures that the samples fracture. The second method involves dropping free falling weights onto samples. Free falling drop tests allow higher velocities and impact energies to be achieved. Useful data can also be gathered by doing impact tests at different temperatures as often this reflects more accurately the kind of environmental conditions the plastic will be subject to. For example, how a car bumper performs under impact at 25 ºC may be different to how it performs at -10 ºC. It may become more brittle at low temperatures. Whatever impact test method is employed, polymer degradation should, like tensile testing, show up as a reduction in the property measured. [4]

III. Results and Discussion

All graphs are normalized so that the virgin material is 100%. Other values are scaled to this value.

A. Tensile test

1) Elastic tensile modulus

First very important material parameter is obtained by measuring the tensile modulus E. As can be seen in Fig. 4 virgin polycarbonate has the highest value of elastic modulus. Recycling caused a noticeable reduction of the modulus. This is mostly visible in the mixture of crushed material with particles smaller than 1 mm. Value of the modulus decreased by almost 300 MPa. It is more than 20% in comparison with the virgin polycarbonate.

Table III Elastic modulus

Mixture	**Elastic modulus E [MPa]**	
	Arithmetic mean	Standard deviation
Virgin polycarbonate	1891	147,9
Sieved crushed material	1911	141,5
Non-sieved crushed material	1601	277,5
Crushed material 1-2mm	1638	214,8
Crushed material < 1mm	1475	301,5

Elastic modulus value decreased about 200 MPa in the mixtures of non-sieved crushed material and particle size 1 – 2 mm. That it is about 15 % less than virgin material.

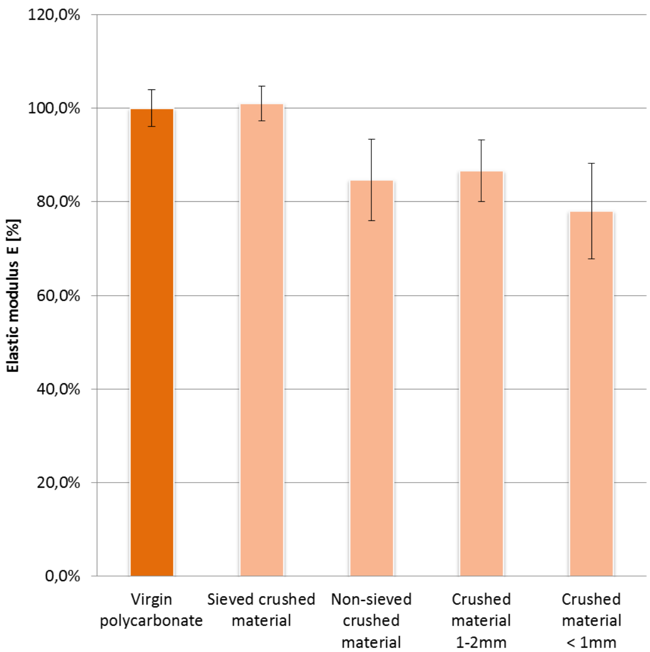

Fig. 4 Elastic modulus

Mixtures containing small particles (non-sieved, 1 < 2 mm and dust mixtures) gained more unstable statistical characteristics, having a larger variance of measured values than a mixture of virgin polycarbonate or sieved crushed material.

2) Ultimate tensile strength

Ultimate tensile strength is another measured material parameter. This parameter will not show as large differences between the recycled mixtures to virgin polycarbonate as the elastic modulus previously mentioned.

Table IV Ultimate tensile strength

Mixture	**Ultimate tensile strength [MPa]**	
	Arithmetic mean	Standard deviation
Virgin polycarbonate	44,7	0,70
Sieved crushed material	43,6	0,59
Non-sieved crushed material	44,7	0,94
Crushed material 1-2mm	43,3	0,59
Crushed material < 1mm	42,9	0,47

All mixtures show small decrease of tensile strength, but only about 3 %. Biggest decrease is observed in mixture with dust particle. Only non-sieved crushed material behaved as the virgin material.

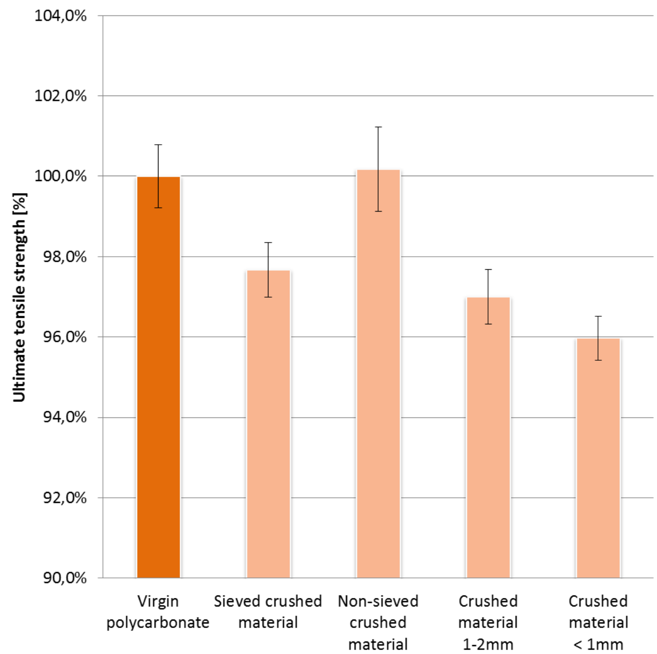

Fig. 5 Ultimate tensile strength

3) Strain

Nominal strain is the last evaluated parameter of the tensile tests. As can be seen from Fig. 6, sieved crushed material elongated less than virgin polycarbonate.

Table V Strain

Mixture	**Strain [%]**	
	Arithmetic mean	Standard deviation
Virgin polycarbonate	5,32	0,19
Sieved crushed material	5,15	0,12
Non-sieved crushed material	5,60	0,59
Crushed material 1-2mm	5,72	0,45
Crushed material < 1mm	5,88	0,61

After recycling all other mixtures elongated more than the original virgin material. The strain values of these mixtures show a similar elongation, about 6% more.

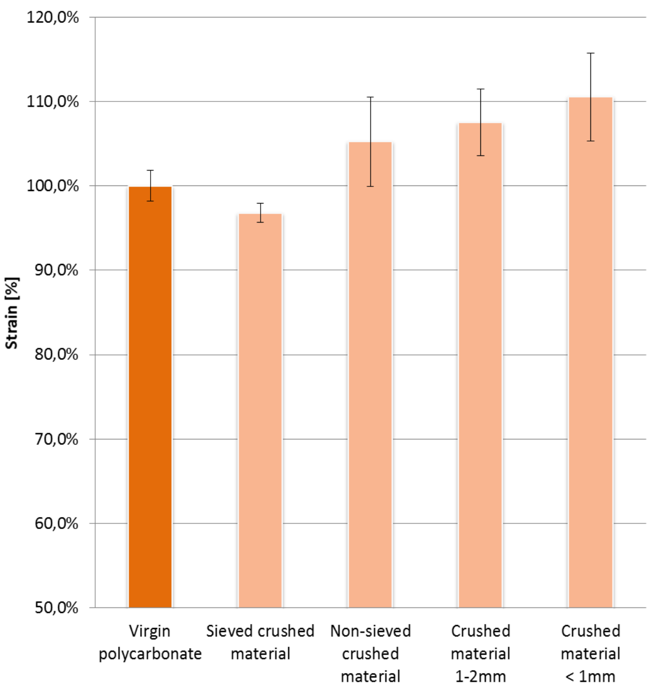

Fig. 6 Strain

Furthermore, the standard deviations of this more elongated mixtures is bigger. Overall, these differences are relative small (maximum strain is 10% more than virgin PC) and they show a tendency of the effect of recycling to relative strain values.

B. *Charpy impact test*

1) *Charpy notch toughness*

Graf of notch toughness at 100 ° C shows, how individual materials behave when tested. As it can be seen in the picture (Fig. 7), the highest values of toughness are reached with mixtures of PC and non-sieved crushed material. Mixture with non-sieved crushed material behave very similarly to the original material.

Table VI Charpy notch toughness

Mixture	**Charpy notch toughness [kJ.m^{-2}]**	
	Arithmetic mean	Standard deviation
Virgin polycarbonate	11,4	1,14
Sieved crushed material	10,9	1,76
Non-sieved crushed material	11,7	1,24
Crushed material 1-2mm	10,0	1,27
Crushed material < 1mm	10,7	1,58

Small decrease is can be seen at mixture with sieved crushed material. Impact toughness is also relatively equable and dispersions in the order of magnitude of 0,5 kJ.m^{-2}.

At first look, it is apparent that the lowest values reached mixture with particle size 1 to 2 mm. And this drops is approximately 12% than virgin material. Mixture with dust particles is even slightly higher value of toughness than previous mixture.

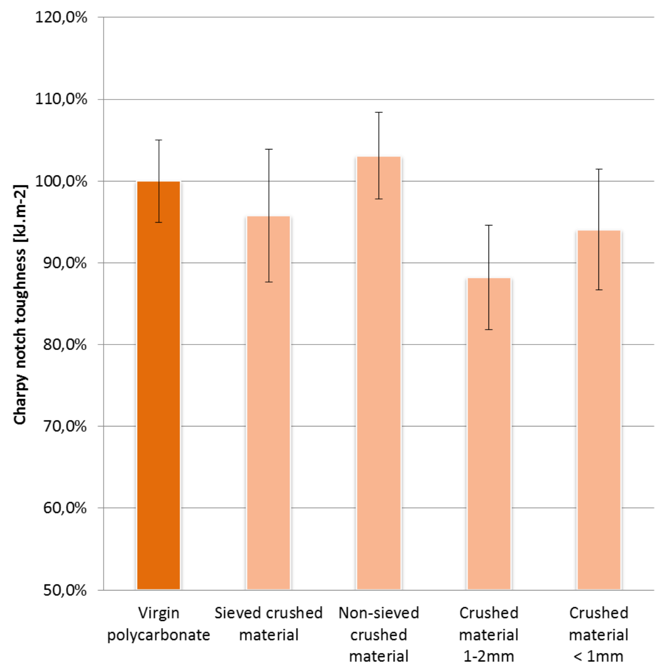

Fig. 7 Charpy notch toughness

2) *Charpy breaking force*

Graf of force required for breaking the sample shows that the size of the forces for each material. The highest force for breaking of the specimen has to be applied in mixtures of virgin PC and sieved crushed material. Values vary in a small range. Smaller standard deviation values come off mixtures of sieved crushed material, and therefore values at this temperature are more uniform.

Table VII Impact breaking force F

Mixture	**F [N]**	
	Arithmetic mean	Standard deviation
Virgin polycarbonate	544	37,7
Sieved crushed material	546	33,4
Non-sieved crushed material	525	34,7
Crushed material 1-2mm	493	34,7
Crushed material < 1mm	475	46,2

For all other mixtures reduction of the force needed to break is 5 to 10 percentage on an average. Only for mixture of smallest dust particles the required force is further decreased by another 5%. The biggest standard deviation there can be seen for this mixture and therefore less stable behaviour in comparison with virgin polymeric material.

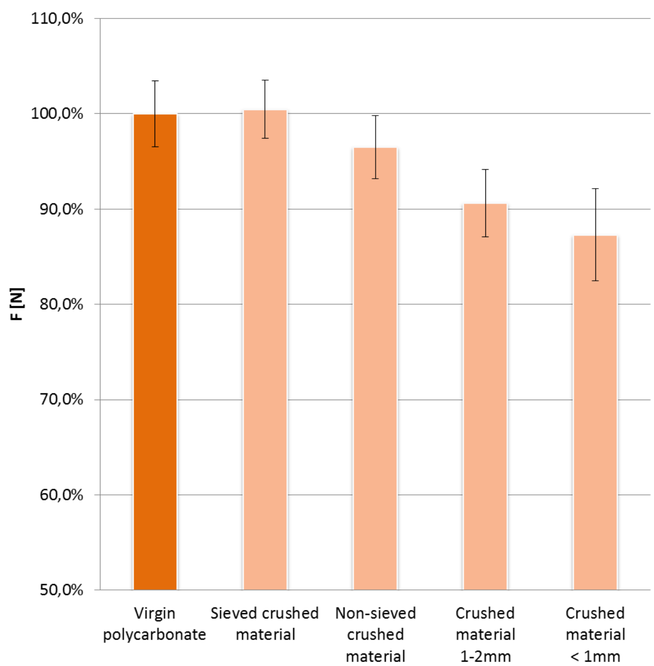

Fig. 8 Breaking force F

C. Hardness test

The last test was a test of hardness Shore D. From measured hardness it is possible to conclude, that particle size of recycled material has little effect on the diversity of hardness values in the case of this experiment.

Table VIII Impact breaking force F

Mixture	Shore D	
	Arithmetic mean	Standard deviation
Virgin polycarbonate	73,64	0,79
Sieved crushed material	71,62	0,53
Non-sieved crushed material	70,97	0,79
Crushed material 1-2mm	70,10	0,99
Crushed material < 1mm	69,30	1,00

It can also be seen, that in the case of virgin PC without recycled material, standard deviation of measured value is small. The other way around, non-sieved crushed material leads to higher standard deviations. For mixtures of PC and sieved crushed material standard deviations are smaller. This suggests that the addition non-sieved crushed material results in more varied material in terms of hardness.

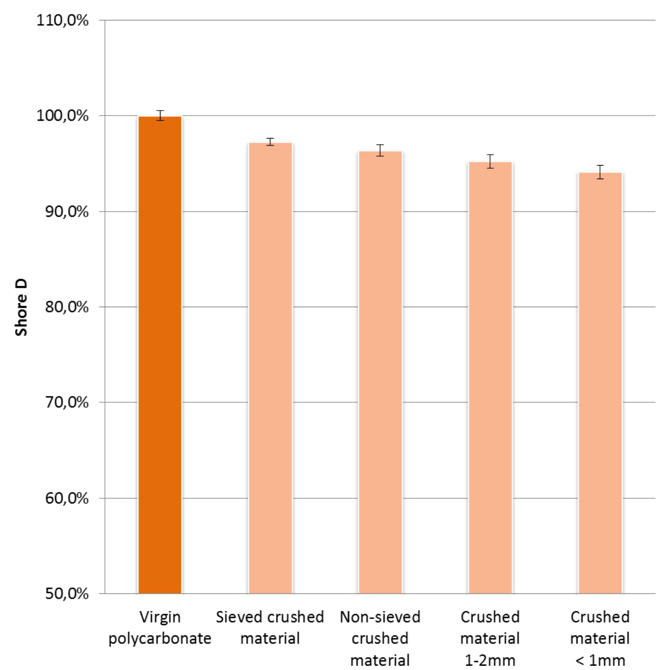

Fig. 9 Hardness Shore D

IV. Conclusion

This work examined the influence of particle size of recycled polymeric material on the mechanical tensile properties of polycarbonate at elevated temperatures. Several mixtures of recycled material were measured. These mixtures differed in particle size after crushing.

Specifically, these mixtures are: non-sieved crushed material - with all crushed sizes of particles; sieved crushed material - without small dust particles smaller than 2 mm, similar to the virgin granules size; then mixture with small (1 - 2 mm) and dust particles (smaller than 1 mm). The measured data were graphically represented and interpreted.

Testing was performed on a universal tensile testing machine. By comparison of the virgin polycarbonate and recycled mixtures we can seen that the effect of the size of the particles was mainly on the elastic modulus. Value of the modulus decreased more than 20 % in mixture with dust particles. For other tensile properties, this effect is not so significant. Values of ultimate tensile strength decreased by 4 %. Mixtures containing small and dust particles are more elongated as the virgin material by about 6%, only sieved crushed mixture showed smaller elongation.

Charpy impact test was next mechanical test, which tested recycled mixtures. Larger decline of notch toughness values occurs in mixtures with small particles (1 - 2 mm) or dust particles. Recycling and hence particle size caused a gradual reduction of the force necessary to break. This decrease is almost 13% to virgin PC, which is already significantly reduction.

The last test was a test of hardness Shore D. From measured hardness it is possible to conclude, that particle size of recycled material has little effect on the diversity of hardness values in the case of sieved or non-sieved mixtures. For the case of dust particles difference of hardness is larger when comparison to virgin material.

Mixtures with dust particles typically indicate higher statistical variations of measured values, and therefore have less stable behaviour of the measured properties compared to virgin polycarbonate, or sieved crushed material. Therefore it is recommended to remove the dust particles when using recycled polymer material. Products made using these recycled materials will have more similar properties as one made with an original material.

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