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Considerations on the Drying of the Raw Material and Consequences on the Quality of the Injected Products

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Abstract. *The first part of the paper presents the specific issues from the injection molding associated with the water content of the hygroscopic plastics (water adsorption, equilibrium moisture level, chemical affinity, hydrolysis) and recommendations regarding the drying of plastics for injection, the drying methods and defects specific to products injected due to the moisture content. The experimental results on the injection of a polyamide (AKULON) and of a thermoplastic polyurethane (DESMOPAN) presented in the second part of the paper are focused on the surface appearance fault "splay" associated with the different values of moisture content for these two materials, verify the value for the admitted moisture content for two hygroscopic materials, polyamide and thermoplastic polyurethane, and ends with conclusions on the residual humidity allowed and opinions on choosing the drying technology and parameters*

Keywords*: plastics, moisture, drying, faults, injected parts, residual moisture content, splay*

1.Introduction

Moisture absorption (also known as water absorption) is the capacity of a material to absorb moisture from its environment. In a typical environment to which polymers are exposed, water molecules may be present in the forms of vapor, liquid or both. Less or more, a polymeric material contains a certain amount of water.

Dehumidifying or drying plastics before processing is a vital part of injection molding process. In the case of polymeric raw material, the purpose is to minimize or eliminate complications that may be caused by too much moisture in a plastic material.

In the machine barrel, even a reduced amount of moisture in the polymeric bulk will turn into steam that mixes with the melt and give, on the injected parts, at least a distinctive fault: splay or "silver streaks".

The extent to which moisture affects the quality of a molded part is determined by the type of polymeric resin being processed, drying time and drying technology.

When assessing the impact of moisture on product quality, it is important to recognize that not all problems are necessarily visible as the silver streaks. Hidden flaws such as reduced mechanical strength can only be discovered using suitable methods of analysis, mostly by means of destructive testing.

On the whole, material with excessively high moisture content, much over an acceptable limit, can negatively impact molded parts in practically every aspect, not only their mechanical properties, but also their optical, chemical and physical characteristics. The most obvious issues include streaks, cavities and holes, blooming appearance and bubbles on the surface, [1-4].

The resin drying prior to processing maintains the performance characteristics of the material in use, avoids supplementary costs by reducing the scrap and, thus, it could be taken into consideration as a strategic concern for maintaining competitiveness.

The goal of this paper is to highlight and to clarify essential details regarding the drying of raw polymeric materials for injection molding, in general, and to verify the value for the admitted moisture content for two hygroscopic materials, polyamide and thermoplastic polyurethane by monitoring the silvers streaks on the injected parts molded from raw material with various moisture content.

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General considerations on the effect of the moisture in the polymer bulk

Exposure to humid air, immersion in water and exposure to boiling water or steam can result in distinctly different material responses. The affinity of a polymer for water molecules could be deducted from the increase in weight percent [% weight gain, w%] of an exposed plastic specimen and can be verified under the following procedures, [5-7]

- water absorption 24 h, at 23°C: immersion of a plastic specimen in distilled water at 23°C, during 24 h;
- water absorption at 100°C : immersion of a plastic specimen in distilled boiling water during 30 min (ISO) or for 2 h (ASTM);
- water absorption at saturation: immersion of a plastic specimen in distilled water at 23°C. Measurement occurs when the polymer does not absorb water anymore;
- water absorption at equilibrium. Plastic specimen is exposed to a humid environment (generally at 50% relative humidity) at a specified temperature (23°C) for 24 h (ISO) or for 48 h (ASTM).

The equilibrium moisture content can be used to compare the amount of water absorbed by different types of plastics when these are exposed to moisture.

In industrial practice, the most convenient are capacitive sensor devices (usually Karl Fischer), that are the most accurate because they will only measure the moisture level in the pallets and are easier to use.

Absorbed water molecules produce polar bonds with the amide groups especially in polyamides. Although in a small amount, in the volume of such a hygroscopic material the presence of water molecules in the volume take up space and displace the polyamide molecules and could change the molecular distribution in the volume or even the chemical formula and these modifications are significant enough to change the rheological properties of these materials, [6, 8, 9].

In the volume of a polymer obtained from polycondensation reactions (such as polyamides and polyesters) with a synthesis reaction involving water formation as reaction product, in the presence of water molecules, different chemical reactions, sometimes irreversible, may occur depending on the relative moisture content:

- with a moisture content lower than that of chemical equilibrium: the water is consumed in post-polycondensation reactions and consequent molecular weight will increase, or
- with a moisture content higher than that of chemical equilibrium: a depolymerization reaction with consequent water consumption and inevitable polymer molecular weight decrease may instead occur.

In both cases, the chemical reactions described above involve a molecular weight change and consequently a change in the viscosity of molten polymer especially at the processing temperature, kinetically favorable to these modifications, [9].

For all forms of polyurethane (PUR), the main pathways of chemical degradation due to the contact with the environment are oxidation and hydrolysis. Scientific results states that polyester PUR is more resistant to hydrolysis than polyether PUR, [9, 10], but this is more susceptible to oxidation, [9, 11], and the unanimous opinion is that, for the both types of PUR, an oxidation reaction requires the presence of moisture. Accelerated aging tests, performed on samples of new PUR-ester in a variety of environmental conditions suggest that hydrolysis rather than oxidation is the dominant mechanism for deterioration and that the rate is slowed at low relative humidity, [8, 9]. The resulting polyurethane would have a substantially reduced molecular weight than the original and would become sticky or even viscous at room temperature [10, 12].

Thus, the presence of the water molecules in the volume of the material could lead to changes in the chemistry and structure of the polymer molecule and this could explain the physical changes ordinarily associated with the aging of plastics (brittleness and weakness, discoloration and blooming) but also the depreciation of the mechanical properties of a moisture sensitive material in contact (even for a short time) with a wet environment.

Polyolefins (grades of PE and PP) and polybutylene contain no chemical bonds that are easily hydrolysable. Hence, they absorb a reduce amount of water and essentially are not affected by the presence of this or by aging in moisture conditions.

Faults on molded plastic parts due to the moisture content and consideration about the drying parameters

Grades of polyamides and PET generally show higher water absorption than other engineering plastics. Furthermore, mild to low water absorption is exhibited by PPS, POM, PA12, PC, ABS and SAN. Plastics with very low water absorption are polymers such as PEEK, PSU, PPSU, PPE, or the hydrophobic polymers such as PE, PP, PS and the most part of fluoropolymers, [3, 6, 9, 13]. The only polymer with zero water absorption is PTFE.

Water molecules inserted between macromolecules could act as a plasticizer but they can also chemically interact with the polymer and both situation lead to the reducing of the glass transition temperature, dimension changes to finished parts but these forms of interaction also could affect the mechanical properties (such as mechanical strength elasticity, tensile strength, impact strength) and electrical insulating characteristics. Hydrolytic degradation plays a part if hydrolysis is the potential key reaction in the breaking of bonds, as in polyesters and polycarbonates and the attack by water may be rapid if the temperature is sufficiently high. The amorphous regions are attacked first and most rapidly, but crystalline regions are not free from attack, [9].

In the barrel of the machine, the excessive moisture content of the material could be partially consumed in chemical reactions as previously shown but the most part of this will be transformed into vapor (steam) that could:

- be pushed into the mold with the melt, and will give plenty of micro cavities in the volume of the molded part,
- cause a segregation of the water-soluble components (for example, an improper, soluble additive) from the volume, [14].

Basically, no degradation of the polymer or imperfections in the molding or extrusion should occur if the moisture content is less than a specified limit (allowed residual moisture, for example.: max. 0.15% for polyamides, Table 1a) and, according to the raw material data sheet, this can be processed without pre-drying when this condition is fulfilled. Nevertheless, the raw material producers recommend a preventive pre-drying in order to overcome the fluctuation in moisture from batch to batch and to have constantly good results. The drying parameters are indicated in the material data sheet in pairs of time-temperature data as in the Table 1. If the drying method is not specified, as in Table 1a, it is considered to be the default, hot air drying. As a general rule, the compliance of the temperature-time values leads to the decrease of the water content below the allowed residual moisture value and it is a first condition for obtaining molded parts without flaws.

Table 1. Example of drying parameters

a. for polyamide PA66 (AKULON S222), [15], [16]				b. for polyarylamide (IXEF-PARA), [17], [18]		
Moisture content, [%]	Temperature, [°C]	Time, [h]		Drying method	Temperature, [°C]	Time, [h]
0.1...0.2 %, and as delivered	80	2...4		hot air	80	12
0.2...0.5	80	4...8		with desiccant	120	4 ÷ 5
other specification: for moisture content ≤ 0.15%, the resin can be molded without pre-drying				no other specification; allowed residual moisture not specified		

It is very important to understand that if the dried raw material is not immediately processed and it is subjected to 50% relative humidity (of the environment) a 0.10% moisture increase could occurs after

2 hours exposure (depending on the material), whereas at 100% relative humidity, this value will be reached in less than 1 hour. Therefore, pellets so exposed should be redried before use.

On the other hand, in order to obtain a lower moisture content (under a specific limit) in a shorter time, the logical first solution should be the increase at maximum of the drying temperature. For this purpose, from the data sheet of the raw material, another parameter could be useful: Vicat softening temperature "T_{VICAT}" or "VST". Thermoplastics do not exhibit an exact melting point that indicates the precise transition from the solid to the liquid state. Instead, there is a gradual softening as the temperature increases. T_{VICAT} reflects the point of softening to be expected when a material is used in an elevated temperature application but also indicate that over this temperature value the material became soft and may be sticky. Consequently, for working with non-sticky granules, the maximum drying temperature allowed in the drying device will be:

$$T_{dry\ MAX} = T_{VICAT} - 10\,^{\circ}C, \qquad (1)$$

Of course, using an advanced dryer, technologically designed to speed up water extraction, the drying time will be shorter and this may be a convenient solution for materials with long drying time, Table 1b. So that, in the case of polyamides grades and for polymers with esteric fraction (including the polyester elastomers), for good quality results and shorter drying cycle time, the dehumidified air ovens (with desiccant) are recommended.

For polyamides, polycarbonates, acrylics and different grades of polyester (including elastomers) processed by injection or extrusion, at the temperatures substantially above the melting point, excess moisture causes hydrolytic degradation of the polymer, a loss in physical properties and brittleness of the material, [19, 20]. Perhaps no visual defects will be apparent but poor in-service mechanical performance can occur, particularly at low temperatures in use and the quality concerns are intimate correlated with the affinity of the material for water and the interaction mechanism of the macromolecule with this, Table 2.

Table 2. Polymer interaction with moisture and quality concerns (processed information from [5, 6, 13, 21])

mainly quality concerns to be considered		
no concerns, for hydrophobic materials	visual defects (surface quality, aesthetic appearance), if mild to moderately hygroscopic polymer is used	decrease the performance for which the material was selected, if this is highly hygroscopic
PE PP PS butadiene-styrene copolymer polymethyl-pentene	terpolymers and copolymers of styrene (ABS, SAN, e.a) grades of acrylics grades of acetals PPE/HIPS PPS PVC	PET polyester PC grades of polyamides PBT polyester polyurethane PEI PAI

In the automotive industry are frequent such cases when quality requirements are not limited to visual aspects and the product is subject to compliance tests. When manufacturing these products, the admissible moisture limits should be reconsidered at lower values to ensure that not only aesthetic requirements are met but also the performances of the material are preserved. These assumptions are verified in experimental tests and indicative values for the percentage by weight of permissible moisture in some selected polymer resins for such cases is shown in Table 3.

Table 3. Percentage by weight of permissible moisture in some selected polymer resins, [6], [22]*

material type	permissible moisture, [%]		drying temperature, [°C]
	at injection	at extrusion	
ABS	0.10...0.20	0.03...0.05	75...90
acrylics	0.02...0.1	0.02...0.04	70...80
ethyl cellulose	max. 0.1	max. 0.04	75...90
polyamide	**0.04...0.08**	0.02...0.06	80
polycarbonate	max. 0.02	max. 0.02	120
low density polyethylene	0.05...0.1	0.03...0.05	70...80
high density polyethylene	0.05...0.1	0.03...0.05	70...105
polypropylene	max. 0.05	0.03...0.1	70...95
polystyrene	max. 0.01	max. 0.04	70...80
PVC	max. 0.08	max. 0.08	60...85
* TPU (ether- and ester-based)	**max. 0.05**	max. 0.05	80...110

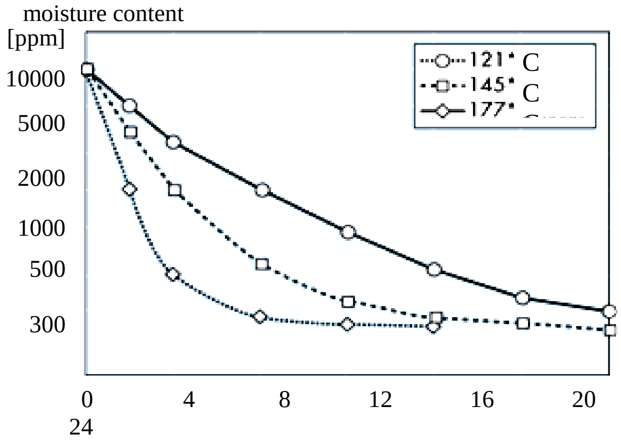


Fig. 1 Evolution of the moisture content on drying PAI with a desiccant drying system

Whatever the polymeric material and the drying technology used, the loss in moisture does not have a linear but asymptomatic evolution, Figure 1. This is so-called "allowed residual moisture", at a value that is not detrimental to the quality of the product, Table 3, and up to which drying is economically justified.

The drying temperature plays an important role in shortening the drying time as shown in Figure 1.

Dryers for raw granular plastics

Drying of granulated plastic for injection or extrusion is applied in two situations:

- a hydrophilic material is used and drying is a technological necessity,
- for the final product, the quality requirements are rigorous in aesthetic or mechanical strength, and even if the material in use is not hydrophilic, removal of residual moisture is a solution for reducing the rate of noncompliance at mechanical tests.

For many years, the choice was simply between hot-air and dehumidifying desiccant dryers and the task was limited on taking into account the materials you needed to dry and on choosing the correct volume of the dryer according to the consumption of material in the process.

Hot-air dryers are the oldest, simplest, and least costly drying technology using a stream of heated ambient air to remove the surface moisture from condensation (if the material was exposed to high humidity and changing temperatures) and less for extracting moisture from the bulk of the raw material, [24]. These are proper to use for non-hygroscopic resins, such as grades of polyolefin (PE, PP), polystyrenes (PS) and PVC, but also to pre-heat materials for injection and extrusion or for drying some mildly hygroscopic materials such as certain thermoplastic elastomers (TPEs) and even acrylics (POM) for non-critical applications, [5, 13, 21].

Often with a brief endowment consisting of no more than a blower, a heater, and a temperature control system, improved sometimes with a vacuum pump on the top of the unit, these hot-air driers are working with ambient air whose moisture charge in rainy days can significantly reduce the efficiency of the device.

Newer versions of hot-air dryers, for small to medium-sized molding machines, are using either heater bands or heat pipes to distribute the heat rapidly and radiant in the hopper at a temperature gradient less ±1° C from top to bottom.

Desiccant dryers. Nowadays accounting for at least 80% of those in use, these dryers are more expensive than hot-air types but for a long time were the best choice for drying hygroscopic plastics whose chemical structure has a strong affinity for water.

The use of the desiccant from the two or more beds accelerates the extraction of moisture from the volume of the processed material, and generally for the same drying results, the residence time in such a dryer is generally at a half of the amount required for using a hot-air dryer. The continuous desiccant dryers with a "honeycomb" rotor using radiant heat technology are claimed to have greater drying capacity than standard hot-air units. For desiccant bed dryers, the drying residence time is typically from 4 to 6 hours, depending on the resin, but desiccant with rotor can dry the same material in 1 to 2 h.

The interest in improving the performance of these dryers has brought to market dryers with straight compressed-air, compressed-air units with a moisture-removal membrane, and vacuum dryers, [11], [24].

The most of these newer types use physical principles that make more efficient the extraction of the moisture, have better energy efficiency but also have size restrictions that could limit their ability to compete with desiccant dryers in high-volume applications and/or for high hygroscopic resins like PET and polyamides grades.

Compressed-air dryer. In these devices, the compressed air introduced at the bottom expands to atmospheric pressure in the drum and drops the Dew point by 10°C. The air is then heated to raise its drying capacity further before it passes up through the resin hopper. This type of dryer has no desiccant, no moving parts, no regeneration heater, no valves or cooling coils and requires a minimal maintenance for the air filter, [6, 11, 24].

It is recommended using them for drying certain hygroscopic resins such as ABS, acrylics, acetals, and glass-filled PA but also for materials that require a lower Dew point, such as PET, PBT, or PC. For these three lasts, the recommendations are to use an add-on desiccant-bed kit or a moisture-removal membrane.

Basically, there is no big difference in energy costs between using compressed air and desiccant drying, the energy used to regenerate desiccant balancing the energy used to compress air.

At the larger models, the configuration of the dryer minimizes energy consumption by separating resin preheating from moisture removal.

Compressed air dryer with membrane also use heated compressed air but also have special moisture-removal membranes that purge themselves automatically and continuously. Using membranes and a refrigeration system, these dryers are able to achieve lower Dew points, so they can dry even the most hygroscopic resins, and could compensate a poor compressor performance but as disadvantage it have to be mentioned the limited size of available membranes, [11, 24, 25].

Vacuum drying. Instead of blowing hot air, the vacuum is use to decrease the boiling point of water and for forcing the extraction of the water vapor from the processed material. This technology permit much lower drying temperatures (for PET, with 20...30°C below the normal drying temperature), eliminates regeneration heating and cooling, use up to 70% less energy than desiccant drying, needs a minimal maintenance and can reportedly dry the most hygroscopic resins, [11, 24, 25].

Just because the plastic raw material is passed through the dryer device at the recommended temperature and length of time doesn’t mean the material is dry enough to process. More than that, it is very important to respect the recommended drying time that have to be the residence time in the drying device, to know the specifications of the device and to understand the drying process.

The lack of information and knowledge about the processed material, the mold in use, the injection machine and the peripherals, any carelessness in organizing the production process can lead to undesirable results. For example, if the desiccant dryer has not been maintained properly (see the maintenance specifications), the device is not working properly, the plastic material that enter into the barrel may still have too much moisture and it may need to be dried for a longer period of time.

2.Materials and methods

In order to obtain the moisture loss curves, to verify the drying efficiency of two hydrophilic materials using a hot air dryer and to find the value of the allowed residual moisture for the material in use, an experimental program was designed and applied for the injection of raw material with different moisture content. The materials used in the study were:

- a polyamide PA66 AKULON S222, drying conditions: T = 80°C and t = 2 up to 4 hours,

packaged at a moisture level ≤ 0.15 w% (according to the material data sheet, [15]), allowed residual moisture: 0.04...0.08 %, according to the Table 3.

- thermoplastic polyurethane DESMOPAN 1050D, injection molding grade; hydrolysis-stabilized,

recommended maximum drying temperature : T = 100°C, [19], [22], allowed residual moisture: 0.05 %, according to the Table 3.

The inspection of the injected products was summarized to the video evaluation of the surface of the injected product and the determination of the intensity of the splay flaw, the first one that occur when in the material is charged with moisture over the allowed limit. Because the splay appears like silver streaks, in order to have in the analyzed photos the contrast convenient for further evaluation, both materials were injected with a proper black pigment charge.

The materials were injected in a four nest mold with polished surfaces at A level (SPI specifications, Ra = about 1 µm), for a plane rectangular (85x60 mm, thickness 1.2 mm) cap with a slight border, that requires a 41 ccm injection shot weight at every 60 seconds cycle time.

The injection machine was an ENGEL VC 330/90 with the maximum injection volume at 150 ccm, with a 20 liters simply hot-air flow dryer. The quality of the material was not affected by the residence time in the injection machine barrel.

Before the experiments, for each type of material, the correct injection parameters were established for raw material subjected at a complete drying procedure, for no flaw parts, and these parameters were maintained at each next procedure with partially dried material.

The dryer was fully loaded with material (about 14 kilograms of material). Each procedure was perform after a residence time in the dryer established in the experimental design (60, 90, ... up to 270 minutes, see the table from the next chapter). With 10 minutes before the end of this residence time in the dryer, the machine barrel was purged and reloaded with new dried material, then 8 consecutive injections were performed in order to reach the optimal temperature, stabilized, for the mold. The parts resulted from the next 2 or 3 injection were withhold and considered appropriate to be subject to the video inspection.

The moisture level was measured before the injection, with a handheld device with capacitive sensor.

The image (JPG format) of the exterior surface of the representative injected parts from each procedure, with a convenient contrast, were processed as shown in Figure 2, by turning it into a negative with the software TOUPVIEW 3.7 (auxiliary at the video camera) and on this, with a metallurgical analysis software (auxiliary at the microscope LM-302) the intensity of the fault splay (silver streaks), in percents, was evaluated.

3.Experimental results

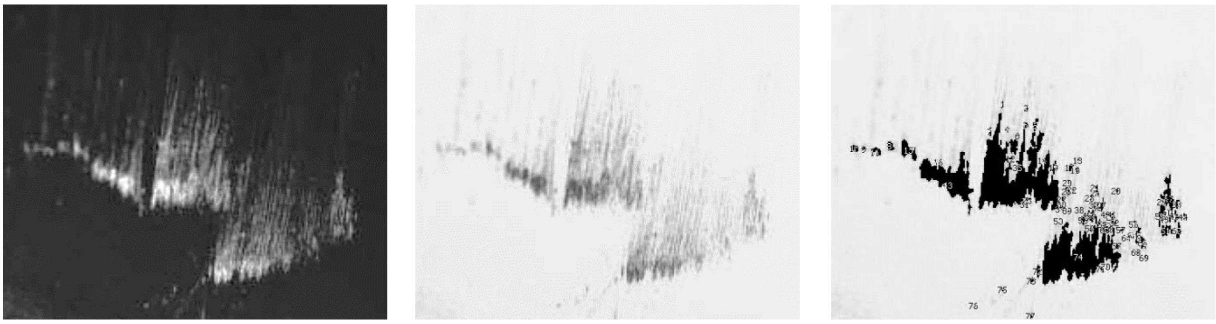
The results of the experimental procedures, each of them with different drying time and for processing condition described above, are summarized in Tables 4 and 5.

Table 4. Injection of polyamide PA66 with different drying time

Material	drying time t , [minutes]	drying temperature, T, [^{0}C]	measured moisture level [w%]	surface appearance after injection
polyamide PA66, AKULON S222	0	---	0.2	splay (silver streaks) on all the surface of the part
	60	80	0.138	
	90	80	0.12	
	120	80	0.1	
	150	80	0.088	splay on 70% from the part surface
	180	80	0.078	
	195	80	0.074	splay on 25% from the part surface
	210	80	0.07	
	225	80	0.067	splay on 5% from the part surface
	240	80	0.065	good surface appearance
	255	80	0.063	good surface appearance
	270	80	0.061	good surface appearance

Table 5. Injection of thermoplastic polyurethane with different drying time

Material	drying time t , [minutes]	drying temperature, T, [^{0}C]	measured moisture level [w%]	surface appearance after injection
thermoplastic polyurethane, DESMOPAN 1050D	0	---	0.21	splay on all the surface of the part
	60	100	0.15	
	90	100	0.129	
	120	100	0.106	
	150	100	0.09	splay on 65% from the part surface
	180	100	0.075	
	195	100	0.072	splay on 15% from the part surface
	210	100	0.068	splay on 5% from the part surface
	225	100	0.063	good surface appearance
	240	100	0.061	good surface appearance
	255	100	0.06	good surface appearance



a.original photo

b. inverted photo (negative)

c. evaluation of the fault intensity, with metallurgical analysis software

Figure 2. Steps in evaluating the intensity of splay faults on injected parts

The moisture loss curves, with the data from the Table 4 and Table 5, are presented in Figure 3.

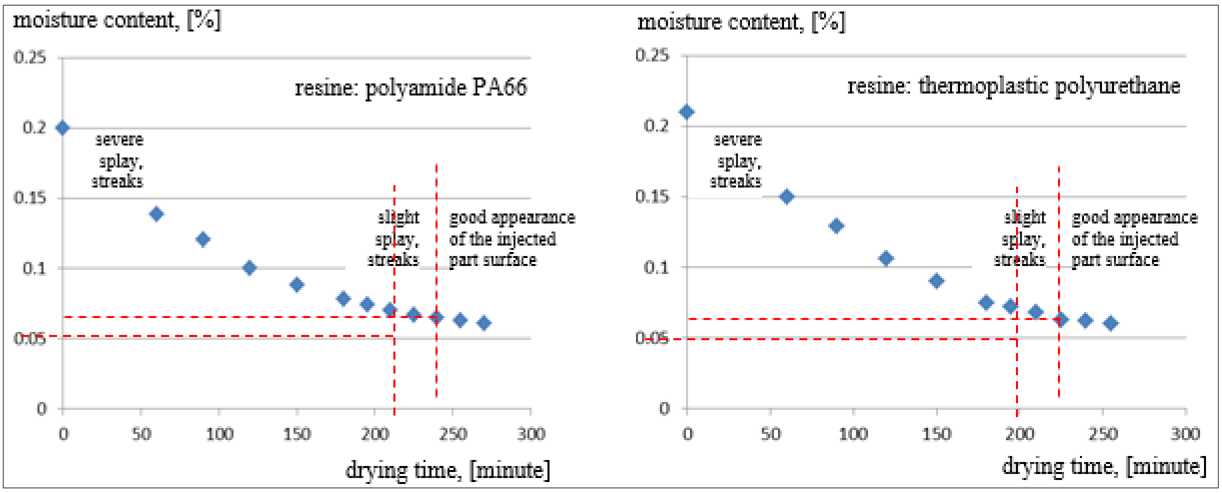


Figure 3. Decrease of the moisture content with the drying time for the materials in the study

4.Conclusions

Exposure to environmental moisture can result in distinctly different responses of the polymeric material according to the affinity of the macromolecule for water molecules (hydrophobic v. hydrophilic).

For both materials used in the study, polyamide and thermoplastic polyurethane, products without surface defects were obtained for residual moisture below the value of 0.065 w%, after a drying time of about 4 hours (240 min). These results match the data from the Table 3 regarding the permissible moisture but the experimental drying time, up to 4 hours, is at the maximum indicated by the raw material producer. This shows the low efficiency of a hot air device for drying hydrophilic raw material.

For each type of polymer there is a specific value of the percentage by weight of permissible moisture in the volume of the resins, the so-called "allowed residual moisture", that is not detrimental to the quality of the product, Figure 1, Table 3, figure 3, and up to which drying is economically justified. A convenient value of moisture content, under 0.05 w%, for both materials, could be obtained in a shorter time by using desiccant dryers or a more advanced drying technology (with compressed air or vacuum).

Frequently, in the automotive industry the quality requirements are not limited to visual aspects and the product is subject to compliance tests. When manufacturing these products, the admissible moisture limits should be reconsidered at lower values from the Table 3 in order to ensure that not only the aesthetic compliance but also to preserve the performances of the material (mechanical, electrical, chemical resistance, ea.).

For both materials in the study, the curves show an asymptotic evolution toward a residual moisture value of 0.05w% which can no longer be reduced whatever long the drying time will be.

The drying parameters are indicated in the material data sheet in time-temperature data pairs (t and T), each parameter can take values within a limited range, from a minimum to a maximum value: from $t_{min}$to t_{max} and from T_{min} to T_{max}. For economic efficiency and faster drying, the first logical solution is to maximize the drying temperature and the highest allowed value could be deducted from the VICAT temperature, information available in the material data sheet, equation (1).

Very important is to choose a dryer with a drying technology adapted to the processed material (hydrophobic/hydrophilic) and having a drying capacity adapted to the feed rate of the process (see the injection shot weight and the cycle time). The residence time in the device will be the effective drying time and have to ensure the moisture removal until the admitted value.

Faults related to surface quality and aesthetic appearance of the part can be easily diagnosed by visual or video inspection but the defects induced by the presence and the action of the water in the volume of the plastic material and that affect the performance of the material can be highlighted only by specific tests.

The drying of the raw material at injection and extrusion is a management task the have to meet technological (directly linked with the quality) but also economical requirements (linked with efficiency and competitivity) both being important for the final result.

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