Report from experimental demonstration carried out as part of the project named *Development of an innovative evolution technique based method for designing dry ice extrusion dies to improve the process efficiency in terms of electrical power and raw material consumption (Contract No.* **LIDER/3/0006/L-11/19/NCBR/2020**), funded by the **National Centre for Research and Development of Poland** under the **LIDER** research scheme

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1. Study method

1.1. General assumptions

The purpose of this research study was to relate the material and energy efficiency of a specific dry ice pelletizer, Triventek PE80, to the geometric parameters of the extrusion die fitted on the machine.

The results obtained with the standard single-hole die supplied by the manufacturer were used as a reference. They were compared with the results obtained with other single-hole dies designed for the purposes of this research. An AI-based method developed for designing single-hole dies using evolutionary algorithms was used for this purpose.

Consumption of liquid carbon dioxide and electrical power used to produce 1 kg of 16 mm dia. pellets were used to evaluate the production process efficiency.

The assumption made at the design stage of this research was that the designed dies, while lowering the consumption of the raw material and electrical energy, should still produce pellets of comparable quality, assessed on the basis of density, being a quantifiable end-product parameter.

1.2. Measurement of consumption of electrical energy and liquid carbon dioxide

The methodology used in this research allows comparison of dry ice extrusion efficiency indicators between different dies of the same inlet and outlet diameters and the overall extrusion cavity length. In the statistical analysis of the results, it is expedient to adopt as the baseline the results obtained with the standard factory-fitted extrusion die. consumption of electrical energy P_C in Wh, consumption of raw material represented as the weight of liquid carbon dioxide used in the process m_{LCO_2} and also the produced pelled weight m_P and density ρ_P. In this way, it was possible to calculate the process efficiency indices for consumption of electrical energy η_E and liquid carbon dioxide η_{RM} per unit weight of produced pellets. Their values were calculated using the following equations (1) and (2).

$$\eta_E = \frac{P_C - P_{C.0} \cdot t}{m_P} \left[{}^{Wh}/_{kg}\right], \quad (1)$$

Umowa realizowana w ramach projektu pt. *Opracowanie innowacyjnej metody wykorzystującej technikę ewolucyjną do projektowania matryc kształtujących stosowanych w procesie wytłaczania skrystalizowanego CO_2 w celu zmniejszenia zużycia energii elektrycznej i surowca* (nr umowy: **LIDER/3/0006/L-11/19/NCBR/2020**), finansowanym przez **Narodowe centrum Badań i Rozwoju** w ramach programu LIDER.

where: $P_{C.0}$ is the average amount of energy consumed in one second of idle run of the machine and t is the duration of test.

$$\eta_{RM} = \frac{m_{LCO_2}}{m_P}\ [-], \tag{2}$$

The test set-up, as shown in Fig. 1 below, consisted of PE80 pelletizer, electricity meter (Fig. 1, label 9), a liquid CO_2 flow meter (Fig. 5, label 8), weighing scale for measuring the weight of produced pellets (Fig. 1, label 10) and an hydrostatic density measurement station (Fig. 5, label 11). In addition, the pressure value of LCO_2 was read and used to calculate the weight of used raw material based on the measured volume.

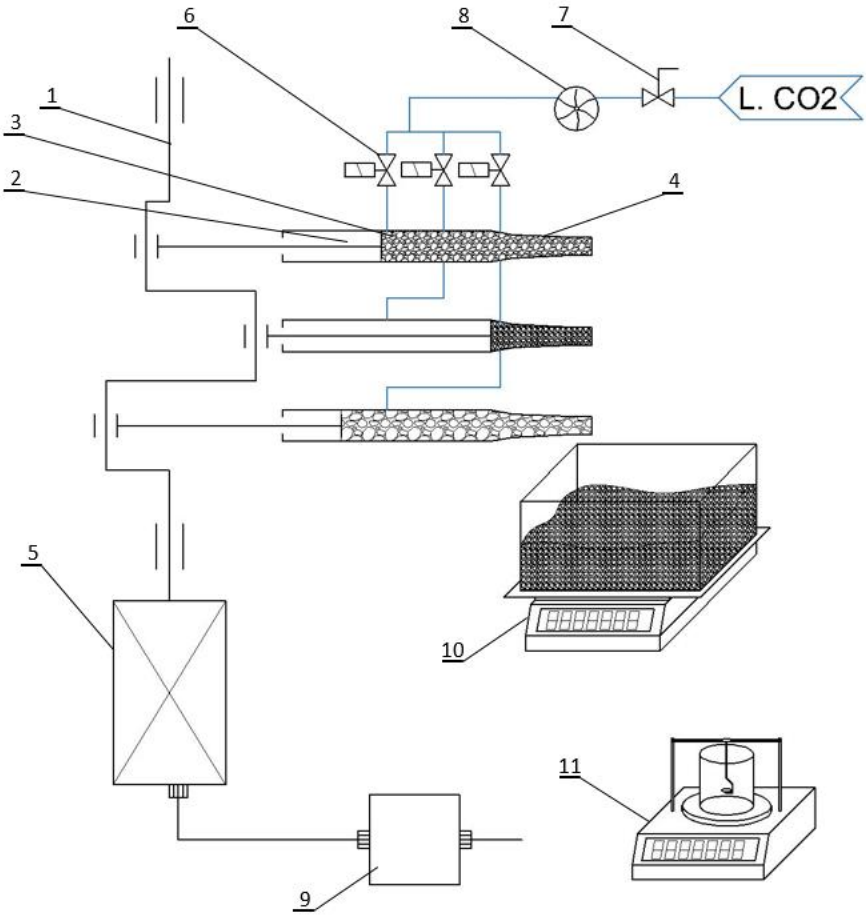


Fig. 1. Schematic of the test set-up for testing the efficiency of dry ice pelletization process using the crank and ram technology. 1 – crankshaft, 2 – ram, 3 – compaction cavity, 4 – die, 5 – 3-phase gear motor, 6 – liquid CO_2 injection solenoid valve, 7 – manual shutoff valve for liquid CO_2, 8 – liquid CO_2 flow meter, 9 – electricity meter, 10 – weighing scale for measuring the weight of produced pellets, 11 – hydrostatic density measurement station.

Before the test, the dies (Fig. 1, label 4) that close the compaction cavity (Fig. 1, label 3) were dismantled from the machine. Next the machine was prepared for start-up as per the factory instructions and the instruments were reset. Then the machine was started to perform a normal working cycle. This allowed recording of the electrical energy consumed to overcome various internal resistances. These include friction forces of the sliding components and on bearing units. The test lasted 10 min with the subsequent measurements taken at 30 sec. intervals. The recorded consumption of electricity has been designated $E_{C.0}$. It ranges from 665.2 to 680.3 J/s. The median of the population of results was 670.6 J/s.

Next, the dies were fitted in the working system and prefilled as per the specified procedure. The instruments were reset. The test set-up was now ready to start the solid carbon dioxide (CCD) extrusion process. The instruments were read every five minutes and the readouts were recorded. The values from the instruments were read 16 times for each type of die.

On completion of the pelletiser efficiency test, the density of the pellets produced was measured using the hydrostatic method described in detail in 1.3 below.

The tests were completed within one day, the air temperature and humidity were measured at one minute intervals using Testo 440 multimeter manufactured by Testo Test Sp. z o.o., fitted with humidity and temperature probe, model No. 6369770 (manufactured in Pruszków, Poland) of ±1.3 % RH and ±0.3 K measuring accuracy.

1.3. Pellets density measurement method

As we can read in the literature, hydrostatic method is widely used for measuring the density of yielding materials such as plastics. This is because it does not require determination of the volume of specimens, problematic in this case. ACN220 analytical scale was used, equipped with hydrostatic measurement module (Fig. 2). The tests were carried out in two stages. First the weight of specimen in air was determined, designated m_0. For this purpose the specimen was placed on the upper scale plate (Fig. 2, label 3). Next the specimen was placed on the lower plate (Fig. 2, label 4), immersed in the test liquid (Rys. 2, etykieta 6), isopropyl alcohol in this case. The weight m_1 equal to m_0 less the effect of buoyancy. This method allowed determination of CCD density, here designated ρ_{CCD}.

During the test it was required to maintain the specified liquid temperature T_L. It should be close to the temperature of CCD in order to reduce the sublimation rate of the tested material. For this method it is necessary to know the relationship between the temperature and the density of liquid ρ_L in which the specimen is immersed.

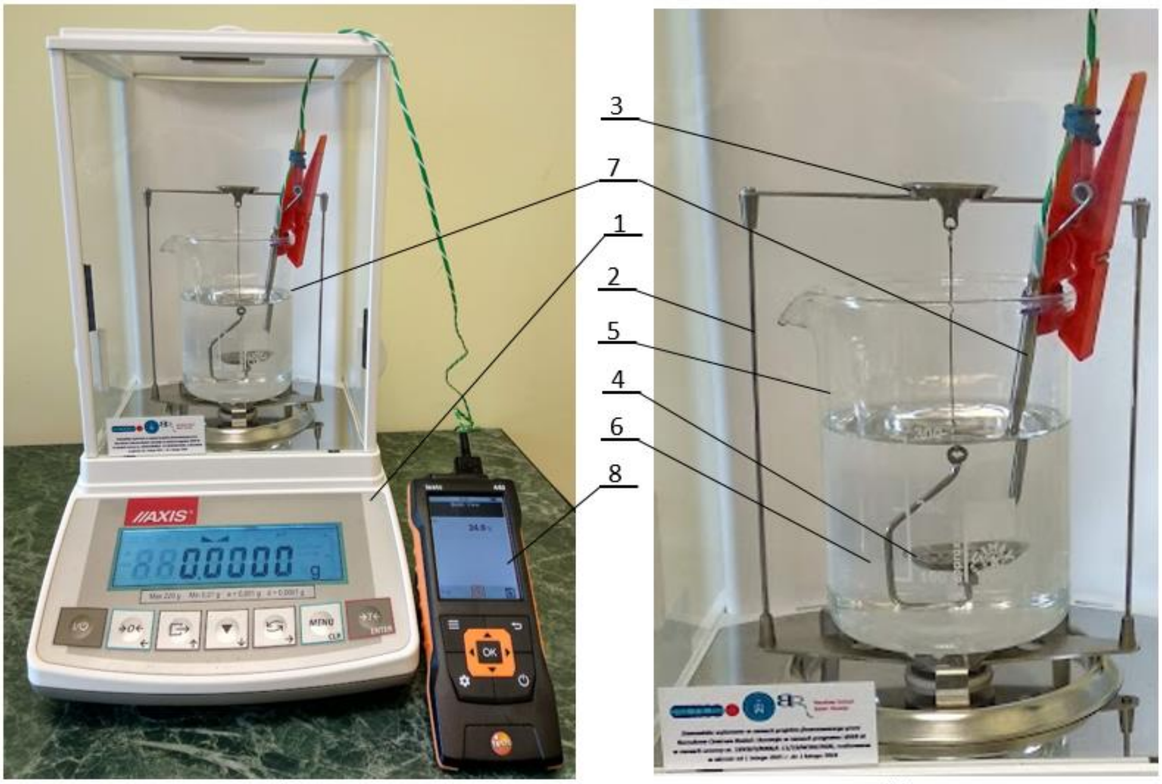
In this case it would be problematic to maintain the liquid temperature T_L at a constant level of 72.5°C (200.65 K), this being much below ambient temperature. For this reason, the following relationship between density and temperature T_L in K was determined experimentally for isopropyl alcohol, i.e. the liquid used in these tests:

$$\rho_{Et}(T_L) = -0{,}947 \cdot T_L + 1{,}07 \cdot 10^3. \tag{3}$$

The liquid temperature was measured to accuracy of ±0.3 K by means of Testo 440 (Fig. 2, label 8) multimeter equipped with a K-type thermocouple (Fig. 2, label 7) manufactured by Test Sp. z o.o. of

Pruszków, Poland. Now the values of m_0 and m_1 could be determined to finally calculate the specimen density with the following equation:

$$\rho_{CCD} = \frac{m_0}{m_0 - m_1} \rho_L(T_L). \tag{4}$$

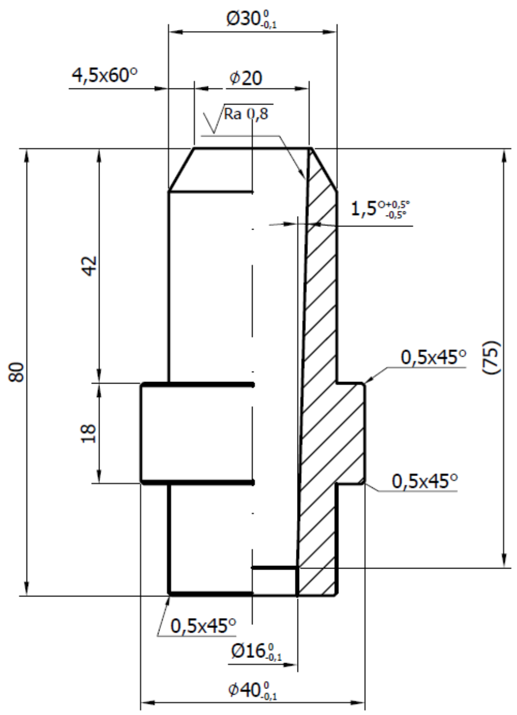


(a) (b)

Fig. 2. FIGURE 5. Hydrostatic density measurement station, a) overview, b) close-up of the hydrostatic test module, 1 – ACN220 scale including draft shield, 2 – hydrostatic measurement system, 3 – upper plate, 4 – lower plate, 5 – beaker, 6 – test liquid, 7 – K-type thermocouple, 8 – Testo 440 multimeter for measuring the temperature

1.4. Geometric parameters of single-hole extrusion dies

Fig. 3 illustrates the geometric parameters of the factory made die supplied as part of delivery of PE80 pelletizer manufactured by Cold Jet. The die cavity is divided into two sections. The first one is conically convergent. It is followed by a constant diameter cylindrical cavity. The population of energy efficiency and end-product density demonstration results for this die has been designated CS.



Rys. 3 Single-hole die with conical/ cylindrical extrusion cavity supplied as a standard accessory with PE80 pelletizer manufactured by Cold Jet.

The next two dies featured modified convergent section of extrusion cavity. The first of them had convex shape and has been designated WP. The geometry of this die is represented in Fig. 4.

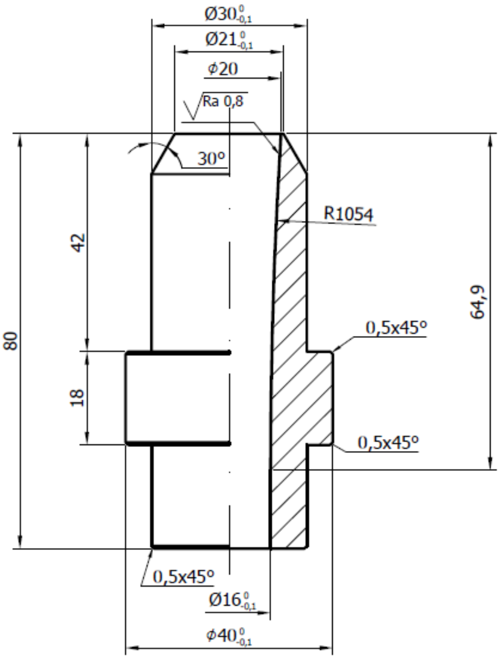


Fig. 4 Single-hole die with convex/ cylindrical extrusion cavity designed for the purposes of this research using the evolution algorithms based design method for designing single-hole dies, developed as part of this project. The shape of the die cavity is protected by UPRP utility model application No. W.131209.

The third die had a concave/ convex convergent section and has been designated WKWP. The geometry of this die is represented in Fig. 5., the same as the previously described dies.

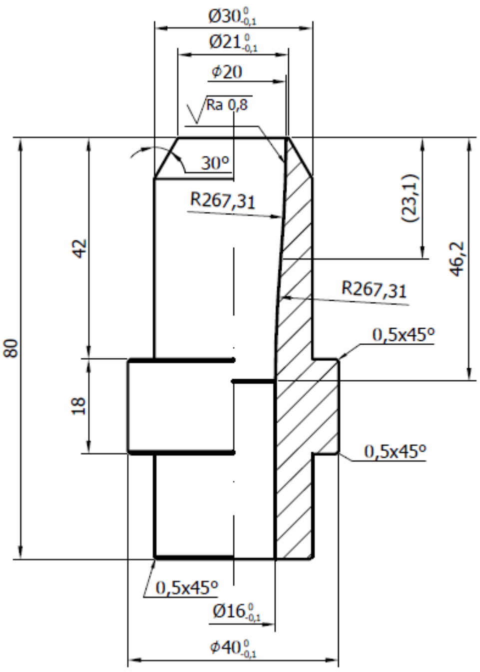


Fig. 5. Single-hole die with concave/ convex cylindrical extrusion cavity designed for the purposes of this research using the evolution algorithms based design method for designing single-hole dies, developed as part of this project. The shape of the die cavity is protected by UPRP utility model application No. W.131208.

1.5. Analysis of results

Three-way ANOVA was used with post-hoc Tukey test for the three populations of results obtained for the three dies under analysis.

The statistical significance of differences in mean density of pellets produced with the three dies was determined as the first part of the statistical analysis. Next the statistical significance of differences between populations in the mean energy consumption per 1 kg of produced pellets was determined. The statistical significance of differences in mean material consumption was checked as the last step of analysis.

Statistica ver. 13.3 program of TIBCO Software Inc. was used for the statistical analysis. All comparisons were made by one-way ANOVA and statistical significance was taken at $p < 0.05$.

2. Results

2.1. Measurement of energy and raw material consumption

The values of E_C, m_p, m_{LCO_2} were recorded 16 times at specified time intervals. Next they were used to calculate the values of η_E and η_{RM}.

In the next step normality of distribution being the initial assumption postulated by ANOVA was tested using the Shapiro-Wilk test. The values of this test parameter for the three populations are given in Table 1, together with the values of η_E and η_{RM}. The obtained values are higher than 0.05 indicating normal distribution of results in the respective populations.

Table 1. Results of Shapiro-Wilk test for energy and material consumption efficiency

	CS	WP	WKWP
η_E	0.0943	0.3361	0.978
η_{RM}	0.6078	0.5929	0.0701

Next Levene's test was used to assess the homogeneity of variance in the populations for each of the analysed efficiency parameters. The obtained values are given in Table 2 below. As it can be seen, both values are lower than 0.05, thus fulfilling the homogeneity of variance assumption.

Table 2. Results of Levene's test for the values of η_E and η_{RM}

	p
η_E	0.036742
η_{RM}	<0.00001

With the initial assumptions fulfilled analysis of variance was performed using the post-hoc Tukey test. The values obtained for η_E and η_{RM} are given Table 3 and Table 4.

Table 3. Statistical significance of comparison of η_E values in the analysed populations

	CS	WP	WKWP
CS	-	0,00012	0,00012
WP	0,00012	-	0,9457
WKWP	0,00012	0,9457	-

Table 4. Statistical significance of comparison of η_{RM} values in the analysed populations

	CS	WP	WKWP
CS	-	0.00105	0.00012
WP	0.00105	-	0.07118
WKWP	0.00012	0.07118	-

These result indicate the probability of error in comparing the data between populations WKWP and WP of more than 5% for both parameters. Considering the assumption made in our method, this precludes comparison of results between populations. On the other hand, the differences between CS and the other populations are statically significant.

Table 5a gives the information required to compare the CS results with the other populations.

Table 5a. Descriptive data of the results of the respective populations

	CS		WP		WKWP	
	η_E [Wh/kg]	η_{RM} [m^3/kg]	η_E [Wh/kg]	η_{RM} [m^3/kg]	η_E [Wh/kg]	η_{RM} [m^3/kg]
Min	16.984	2.324	13.926	1.942	14.429	2.196
Q1	17.05409	2.335	14.000	2.231	14.220	2.104
Q2	17.339	2.356	14.308	2.246	14.574	2.155
Q3	17.857	2.360	14.733	2.262	15.079	2.286
Max	18.413	2.386	15.0	2.324	15.556	2.336
Mean	17.449	2.352	14.441	2.247	14.496	2.190

The mean of results in the respective populations closely approximated the value of median (second quartile Q2). This indicates a high repeatability of the results recorded within the 16 repetitions during which the above-mentioned data were recorded.

With the purpose to simply the calculations to compare the pellet production cost based on energy consumption it was decided to keep [Wh] as the unit rather than converting the values to [J].

Comparing the median values for electricity consumption between CS and WP and between CS and WKWP populations we see that the process carried out with the use of the CS die requires a higher amount of energy to produce 1 kg of pellets. These differences are 3.031 Wh/kg in the case of WP and 2.765 Wh/kg in the case of WKWP.

Table 5b gives the process efficiency results for the three analysed dies. These results indicate a negligibly small increase of the mean efficiency index.

Table 5b. Process efficiency in kg/s

	CS	WP	WKWP
Q2 [kg/s]	0,021	0,022	0,022
Mean [kg/s]	0,020	0,022	0,022

Multiplying the given values by 3600 sec. we get the approximate process efficiency over 1 hour of production. In this case it was Q_m = 79.2 kg/h.

2.2. Density determination

After the above-described process efficiency analysis, the density of the produced pellets was checked. The determination was performed on 16 randomly chosen specimens of pellets produced with the use of CS, WP and WKP dies. Statistical significance relating to mean values obtained for the respective populations was verified by means of ANOVA.

Similarly to the previous analysis, normality hypotheses were verified as the first step. The results of the Shapiro-Wilk test given in Table 6 confirm normality of distribution.

Table 6. Results of Shapiro-Wilk test for electricity and raw material efficiency results

	CS	WP	WKWP
ρ_{CCD}	0.1344	0.8255	0.5730

The value of $p = 0.261906$ in the Levene's test does not fulfil the variance homogeneity requirement. Although the initial assumption was not found to hold true with the post-hoc Tukey, statistical significance value was still determined for comparison of the respective means. The p values were higher than 0.3 indicating lack of statistical significance of the differences between the results. The data characterising the respective values are given in Table 7.

Table 7. Descriptive data of the results of the respective populations

	CS	WP	WKWP
	kg/m^3	kg/m^3	kg/m^3
Min	1,375.58	1,332.78	1,399.21
Q1	1,414.09	1,453.49	1,484.01
Q2	1,471.72	1,490.96	1,512.13
Q3	1,539.20	1,524.08	1,536.26
Max	1,567.03	1,634.03	1,575.38
Mean	1,473.32	1,484.81	1,506.55

The data in Tables 6 and 7 confirm that the density of pellets, chosen as a quantifiable end-product quality indicator remained at a comparable level in all the tests of the demonstration.

2.3. Temperature and humidity measurements

Temperature and humidity of ambient air in the immediate vicinity of the pelletizer were recorded during the demonstration. The recorded values are given in Figures 6-8 and in Tables 8 and 9. The temperature of ambient air may considerably influence the process efficiency. Therefore, when the results of this demonstration are compared with other research projects, attention should be paid to different ambient conditions, beyond control of investigators, that may be the cause of any differences.

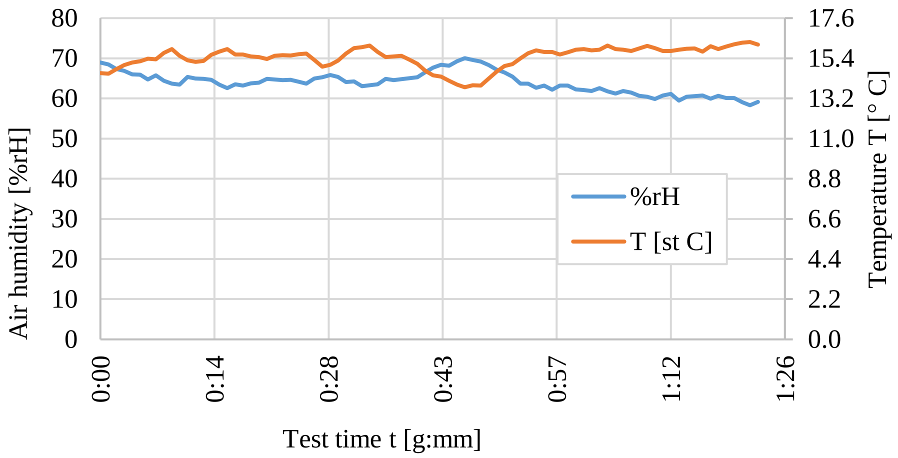


Fig. 6. Variation of humidity and temperature of ambient air during the test with the CS die

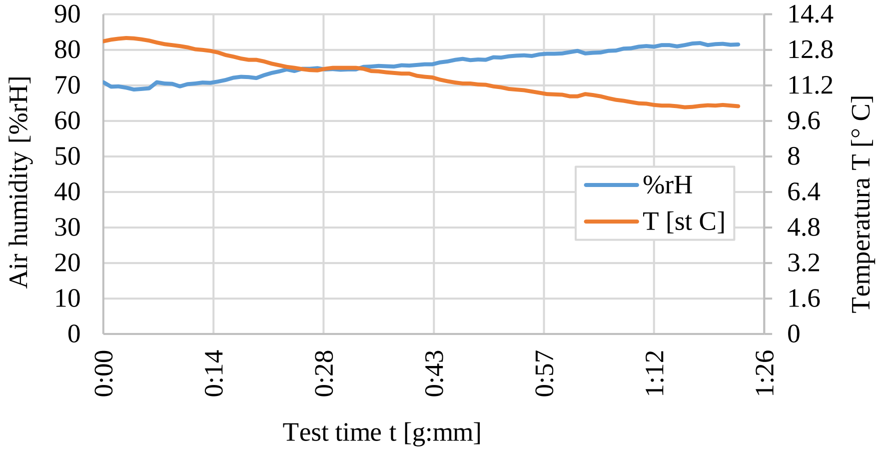


Fig. 7. Variation of humidity and temperature of ambient air during the test with the WP die

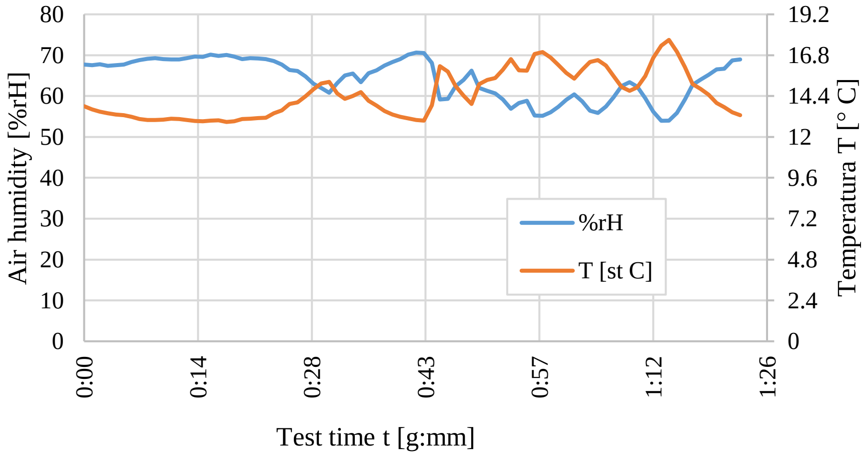


Fig. 8. Variation of humidity and temperature of ambient air during the test with the WKWP die

Table 8. Change of air humidity over time in the respective tests

Die	Min [°C]	Q1[°C]	Q2[°C]	Q3[°C]	Max [°C]
CS	58.3	62.04	63.86	65.36	70.02
WP	68.81	72.75	75.83	79.21	81.89
WKWP	53.96	59.67	65.55	68.86	70.65

Table 9. Change of air temperature over time in the respective tests

Die	Min [°C]	Q1[°C]	Q2[°C]	Q3[°C]	Max [°C]
CS	13.81	15.22	15.61	15.87	16.29
WP	10.21	10.75	11.60	12.29	13.33
WKWP	12.88	13.19	14.18	15.57	17.70

The ambient air temperature around the test stand varied from 10.2°C to 17.7°C and air humidity varied from 54 do 82 %rH. These results were compared with the determined energy consumption and process efficiency indices and temperature was found to have no significant effect in this respect. As mentioned in the method description, the purpose of these measurements was to allow comparison of the obtained experimental results with the results obtained in other research projects. Considering the dependency of the machine efficiency on the ambient conditions it appears indispensable to carry out any future tests in similar conditions. Otherwise, a difference between ambient conditions must be taken into account when drawing conclusions.

3. Final conclusions

The data given in Tables 5a and 5b represent the consumption of electrical power and process efficiency over 1 hour production. This allows determination of approximate consumption of electrical power during 1 hour of regular production, as given in Table 10. The maximum percentage reduction of energy consumption attainable with the designed dies was also determined.

Table 10. Consumption of electrical energy during 1 hour of production

Die	CS	WP	WKWP
Mean consumption of electrical energy [Wh]	1,394	1,154	1,158
Difference [Wh]		240	236
Percent difference in comparison with CS		17.22%	16.93%

As regards the measured consumption of liquid carbon dioxide no significant differences were observed between WP and WKWP dies. This is evidenced by the density of produced pellets of about 1,500 kg/m^3.

As it has been assumed in the project, the material efficiency may reduced if the compression stress exceeds the effective value limit to obtain 1,650 kg/m^3 end-product density. It transpires from the indicated differences that material efficiency depends on the process of expansion rather than extrusion. It must be noted that that percent difference in the consumption of electrical energy depends on the ambient air temperature and humidity around the machine. Therefore, should the ambient conditions change considerably the test results may change accordingly.