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STUDY OF SOME THERMAL PROPERTIES OF HM ADHESIVES*

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For studies were selected 5 HM adhesives with trade name Jowatherm. The softening temperature T_m of adhesives was determined with the use of ring-ball method, Kofler's heating metal, and from thermoanalytic DTA curves. On the base of the data from TG curves were calculated values of E_a of the thermal decomposition process of HM adhesives in the function of conversion using equation of Ozawa-Flynn-Wall.

Key words: HM adhesive, thermal property, softening temperature, TG, DTA, degree of conversion

INTRODUCTION

Technology of gluing up surfaces of board elements as well in the standard system as in the solutions of soft- and postforming, and profile wrappers has reached high level of automatization, mainly due to introduction to the industrial practice of HM adhesives on the base of copolymers EVA. Presently for production of adhesives HM besides copolymers EVA are often used also reactive thermoplastics e.g. PA, polyolefines (APAO) based on atactic PB, PE, PP, and also PUR prepolymers. The glue lines from those adhesives are showing higher thermal durability than from EVA (Anonym 1992, 1993, 1994, Brückner 1993, Schlegel 1993, Seeliger 1993).

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One of the most important thermal properties of HM adhesives is their softening temperature (T_m). This property plays essential role in the processes of application of HM adhesives. It decides among others on flowing index, open assembly time, tackiness, and also on thermo-resistance of obtained glue lines. From the literature results, that in the practice, T_m of HM adhesives is determined by various not correlated measurements methods (Mattern 1991, Proszyk and Bernaczyk 1995, Proszyk, Bernaczyk and Krystofiak 1995, Simon 1996).

Some deficiency evokes also scarcity of the data concerning thermal resistance of HM adhesives, and especially of values of activation energy in course of decomposition process.

In aim to complete mentioned above gaps were undertaken studies, which have in scope to determine of values T_m of HM adhesives basing on various thermoplastics. For this purpose selected measurement methods will be used, and determination of activation energy of thermal decomposition of particular adhesives in the function of conversion degree of this process.

EXPERIMENTS

For studies were selected 5 HM adhesives with trade name Jowatherm (Table 1). The T_m of the adhesives was determined with the use of ring- ball method (acc. to the PN-73/C-04021 standard), Kofler's heating metal, and from thermoanalytic DTA curves. In the method ring- ball the readings of T_m were made in three phases of measurement, which schematically are shown on the Fig. 1. The studies of thermal proper-

Table 1

Tabela 1

Properties of Jowatherm HM adhesives used in investigations

Właściwości klejów HM Jowatherm użytych do badań

Properties Właściwości	Type of Jowatherm adhesives and basic thermoplastic polymers Rodzaj kleju Jowatherm i jego baza polimerowa				
	211 50	227 20	280 70	282 00	288 50
	PA	APAO	EVA	EVA	EVA
Viscosity, mPa·s Lepkość, mPa·s					
180°C	-	-	-	100 000	280 000
190°C	6 000	160 000	-	-	-
200°C	-	120 000	110 000	60 000	130 000
Density, g/cm^3 Gęstość, g/cm^3	0.95[x)]	1.10	0.95[x)]	1.40	1.30

[x)] unfilled adhesive

klej niewypełniony

ties of adhesives were carried out with the use of thermogravimeter Shimadzu TA-501 making measurements in the free air at following parameters:

- mass of samples 15±2 mg
- heating rate 5-20°C/min
- range temperature 20 - 600°C
- standard substance α-Al_2O_3.

RESULTS

On the Fig. 2 and 3 are presented as examples thermograms (DTA-TG) for selected Jowatherm adhesives. From the DTA thermograms was determined T_m of the adhesives reading at the heating rate 5°C/min. Average values of T_m of adhesives obtained with the aid of various measurement methods are listed in the Table 2.

The analysis of the data from the Table 2 gives the proof, that T_m values of HM adhesives are in the considerable degree dependent upon adopted method of measurement and kind of thermoplastics applied for the production of particular adhesives. Evidently upon obtained measurements results could influence also on proper-

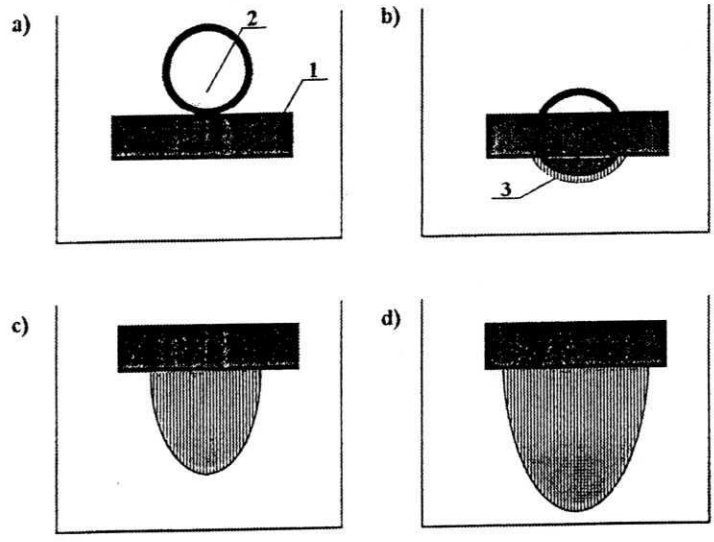

Fig. 1. Scheme of the stages at determination of the softening temperatures HM adhesives by ring-ball method
a) begining, b) initial, c) indirect, d) ending
1 - ring, 2 - ball, 3 - softening adhesive

Rys. 1. Schemat kolejnych etapów przy określaniu temperatury mięknienia niektórych klejów topliwych metodą pierścień-kula
a) etap wyjściowy, b) etap początkowy, c) etap pośredni, d) etap końcowy
1 - pierścień wypełniony zestalonym klejem, 2 - kula, 3 - uplastyczniający się klej

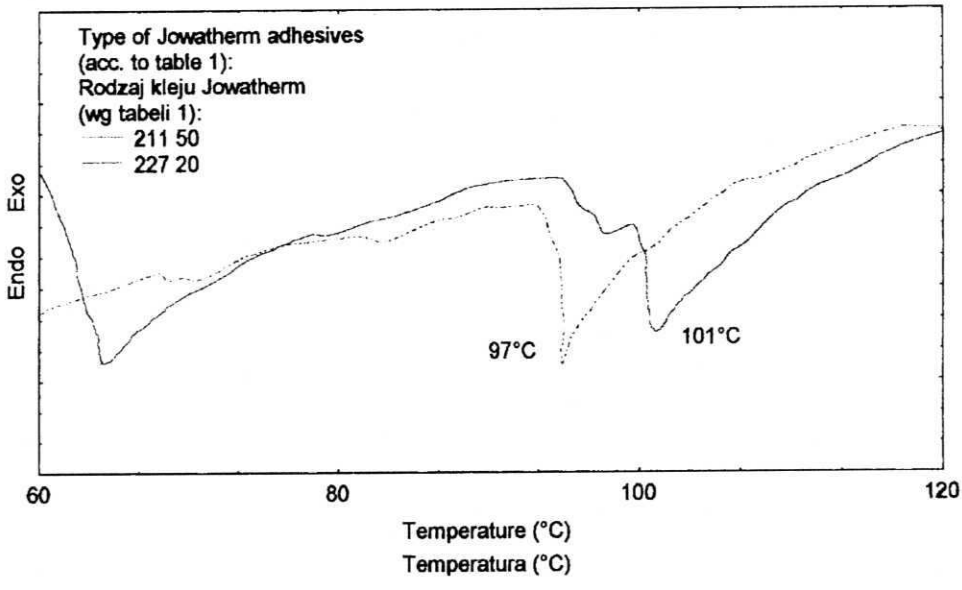

Fig. 2. DTA thermograms for selected Jowatherm adhesives at heating rate 5°C/min
Rys. 2. Termogramy DTA dla wybranych klejów Jowatherm przy szybkości ogrzewania 5°C/min

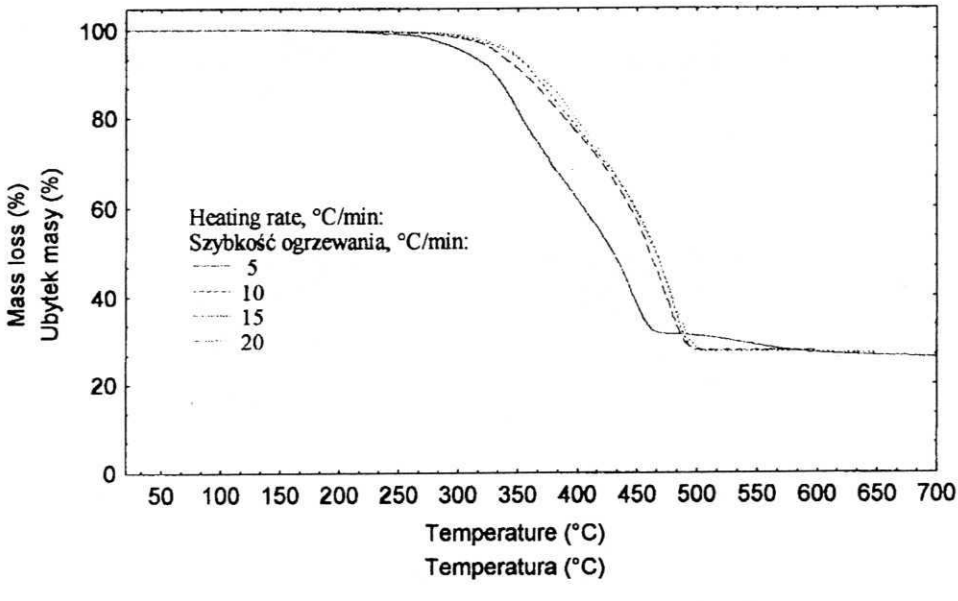

Fig. 3. TG thermograms for Jowatherm 227 20 adhesive at selected heating rate
Rys. 3. Termogramy TG dla kleju Jowatherm 227 20 przy wybranych szybkościach ogrzewania

Table 2

Tabela 2

Values of the softening temperature determined with various methods for Jowatherm HM adhesives

Wartości temperatury mięknienia klejów HM Jowatherm oznaczone różnymi metodami

Type of Jowatherm adhesive (acc.to table1) Rodzaj kleju Jowatherm (wg tabeli 1)	Methods of measurement Metody pomiaru			
	Ring - ball Pierścień - kula		Kofler's heating metal Szyna grzewcza Koflera	DTA
	Measuring stage (acc. to fig. 1) Etap pomiaru (wg rys. 1)	Softening temperature (°C) Temperatura mięknienia (°C)		
211 50	b	125	130 - 140	97
	c	- [1]		
	d	129		
227 20	b	155	160 - 170	101
	c	157		
	d	158		
280 70	b	89	90 - 100	79
	c	94		
	d	101		
282 00	b	78	80 - 90	94
	c	83		
	d	86		
288 50	b	86	95 - 105	65
	c	92		
	d	101		

[1] no this stage
brak tego etapu

ties of other constituents used for production of said adhesives. Accordingly to the expectations the highest values of T_m were characterised HM adhesives on the base of APAO and PA. As it is to be thought, above diversification allows for elaboration of correlation dependencies between particular measurement methods of T_m value.

On the base of the data from TG curves were calculated values of activation energy (E_a) of the thermal of decomposition process of HM adhesives in the function of conversion degree using equation of Ozawa-Flynn -Wall in the form:

$$0.457\frac{-E_a}{R} = \frac{d\ln(\Phi)}{d(T_m^{-1})} \qquad \text{(kJ / mole)}$$

where:

E_a - activation energy

R - gas constant

Φ - heating rate

T_m - temperature in the constant degree of conversion.

When analysing values ln Φ in dependence from T_m^{-1} was obtained straight line with slope $-E_a/R$. In such a way from dependence of decomposition temperature of HM adhesives at given conversion degree from the heating rate of the sample was calculated E_a of studied reactions on the base of dependency:

$$-E_a/R = b$$

where:

b- directional coefficient of straight line.

The courses of dependence ln Φ upon T_m^{-1} for example adhesives are presented on Fig. 4.

On the base of above data were drawn dependencies of E_a changes of the thermal decomposition process of HM adhesives in function of conversion degree. Results of those dependences are presented on Fig. 5 and 6.

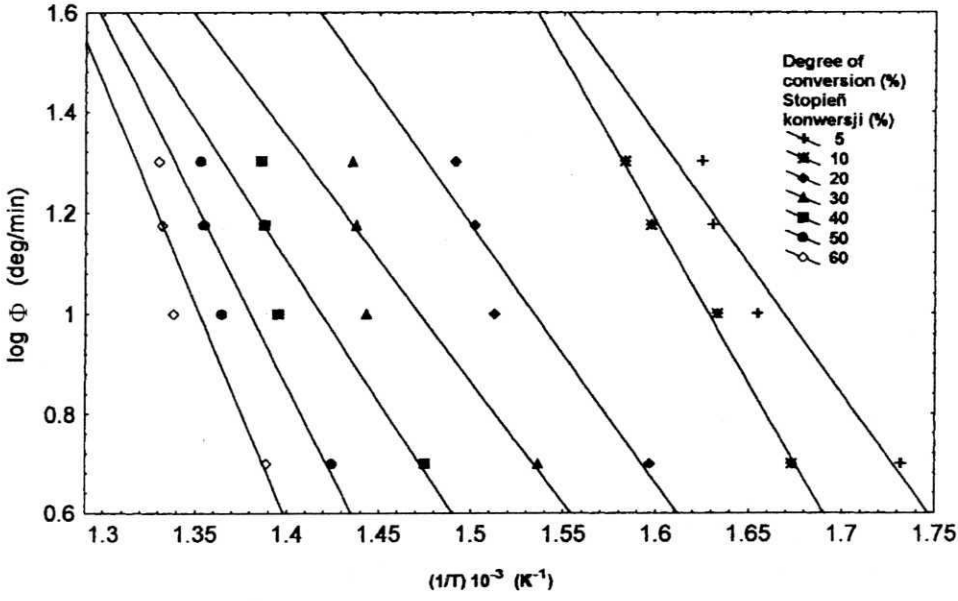

Fig. 4. Plot log (Φ) vs. (1/T) for thermal degradation process of Jowatherm 227 20 adhesive

Rys. 4. Zależność log (Φ) od (1/T) dla procesu termicznej degradacji kleju Jowatherm 227 20

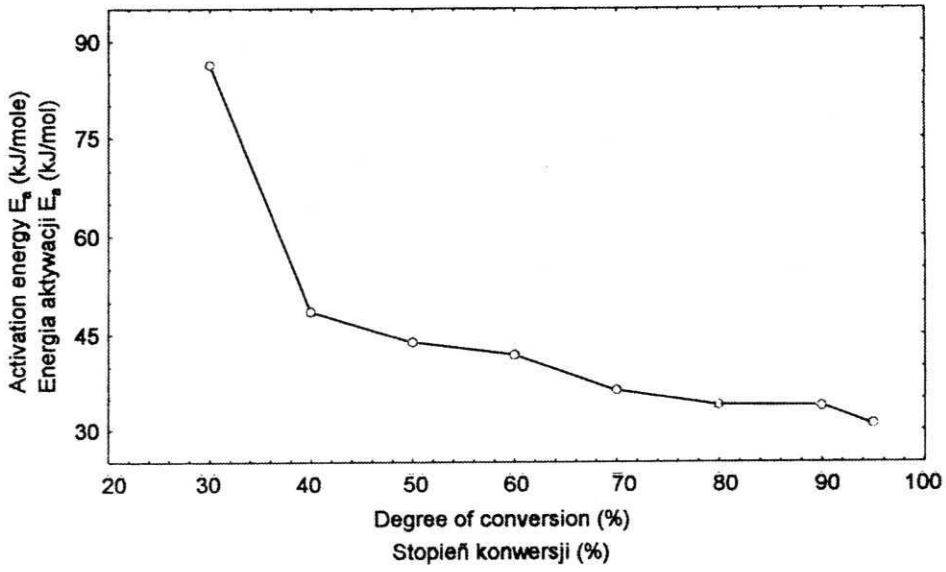

Fig. 5. Changes of activation energy vs. degree of conversion for degradation process of Jowatherm 227 20 adhesive

Rys. 5. Zmiany energii aktywacji w funkcji stopnia konwersji procesu degradacji kleju Jowatherm 227 00

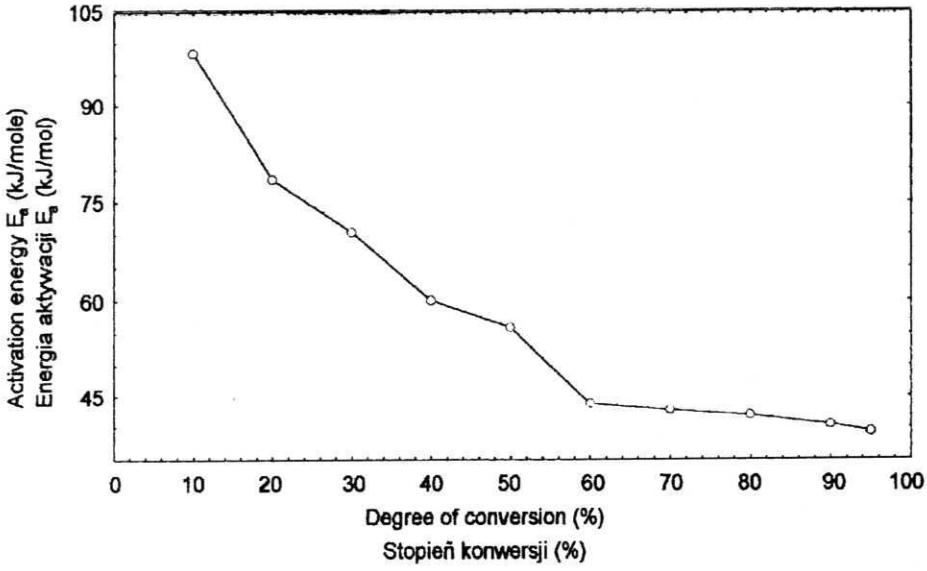

Fig. 6. Changes of activation energy vs. degree of conversion for degradation process of Jowatherm 280 70 adhesive

Rys. 6. Zmiany energii aktywacji w funkcji stopnia konwersji procesu degradacji kleju Jowatherm 280 70

CONCLUSIONS

1. Taken into respect in the studies HM adhesives were characterised by diversified values of softening temperature, and the highest values have shown adhesives on the base of APAO and PA.
2. Together with degree of conversion took place increase of values of the activation energy of the thermal decomposition process of HM adhesives.

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BADANIA WYBRANYCH WŁAŚCIWOŚCI CIEPLNYCH KLEJÓW HM

Streszczenie

Badano wybrane właściwości cieplne istotne z punktu widzenia termoodporności spoin klejów topliwych (HM). Do doświadczeń użyto kleje HM bazujące na polimerach termoplastycznych APAO, EVA i PA. Określono temperaturę mięknienia klejów za pomocą metody pierścień-kula, szyny grzewczej Koflera i danych z termoanalitycznych krzywych DTA. Wyznaczono energię aktywacji procesów termicznego rozkładu klejów HM w funkcji stopnia konwersji, wykonując obliczenia na podstawie założeń podanych przez Ozawę-Flynna-Walla.

Na podstawie wyników przeprowadzonych badań zaprezentowano zależności pomiędzy wartościami T_m uzyskanymi różnymi metodami, stwierdzając, że najwyższą temperaturą mięknienia charakteryzują się kleje na bazie APAO i PA. Procesy termicznego rozkładu uwzględnionych w badaniach klejów przebiegają w funkcji stopnia konwersji przy wyższych wartościach energii aktywacji.

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