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Influence of cleaned granite dust on the properties of silicone pressure-sensitive adhesives

Abstract

The influence of addition cleaned granite dust on self-adhesive products, such as self-adhesive tapes based on silicone pressure-sensitive adhesives was presented. The new way of waste management, is a very important issue in the modern world. The obtained self-adhesive materials were tested both in terms of the properties of adhesive compositions (pot-life test) as well as the influence on the operational parameters of the obtained self-adhesive tapes, such as adhesion, cohesion, stickiness or shrinkage. The obtained results are presented in the paper below.

Keywords: silicone pressure-sensitive adhesives, granite dust, adhesion, SpecSil

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Introduction

Self-adhesive tapes based on silicone pressure-sensitive adhesives are high-quality products used for special applications, usually in conditions where cheaper products such as tapes made of typical polymeric organic adhesives are not sufficient. Due to the adhesive material (self-adhesive silicone adhesive - Si-PSA), the tapes show excellent thermal stability and transparency to UV radiation, excellent electrical properties, high chemical resistance and resistance to weather conditions. Si-PSA adhesives are made of MQ silicone resins and large segments of silane-functional silicone polymers. They show high flexibility of Si–O–Si connections, low intermolecular interaction, low surface tension. Due to the low surface tension, self-adhesive silicone adhesives are easy to spread on the surface of various materials. The tapes are made by crosslinking process - essential/crucial but difficult step influencing the Si-PSA properties, and thus their potential usage process. The crosslinking of Si-PSAs is limited as contain only methyl and phenyl groups. Usually silicone pressure-sensitive adhesives are cross-linked by using organic peroxides at elevated temperature: between 120°C and 150°C. There are also attempts to modify polymers in order to introduce additional functional groups, giving e.g. polymer contain silicon-bonded vinyl (Si-Vi) functional groups.[1–5] In addition, silicone-based pressure sensitive adhesives have aroused increasing interest in the last decade. More and more often, works related to the modification of the adhesive film are devoted to them, especially with the addition of fillers - physical or physicochemical modification.[6–8]

Amongst the available resources, maximum natural resources are believed to be consumed by the industry. With its development, gradually there is scarcity of natural resources day by day. On the other hand the industries are producing a large amount of by-products and wastes in their various processes, which are causing dumping problems. One way to solve these two problems is to find out the possibility of these industrial wastes to reuse in the construction industry. Some of the industrial wastes is granite dust. Alternatives to the use of this waste are being sought, one of them may be using it as a filler in concrete. It has been also proved that the use of granite powder can be used up to 15% as a replacement to sand. It is also beneficial to the mechanical properties which were found to be increasing than control concrete.[9–11] Granite dust can perform a similar function in various types of resins, where as a filler it can affect the properties of the final products, such as castings or pressure-sensitive adhesives.

In this paper influence granite dust on properties of selected silicone pressure-sensitive adhesives were presented. The adhesive tapes obtained for the addition filler to silicone resin were obtained and characterized. Basic properties such as adhesion, cohesion, stickiness and shrinkage have been determined in order to determine the effect of the selected filler on them.

Materials and methods

Materials

Commercial silicone adhesive resin (Q2-7358) a product of Dow Corning (USA) was used as a adhesives. Granite dust obtained from a stone factory (Poland) was used as a natural material to obtain filler. 2,4-Dichlorobenzoyl peroxide (DClBPO) supplied by Peroxid-Chemie (Germany) was used as the crosslinking agent.

Preparation of Si-PSA

The adhesive compositions based on Q2-7358 resin containing different content of granite dust was obtained. Based resin and the crosslinking agent (1.5 wt. % 2,4-dichlorobenzoyl (DClBPO) peroxide according to polymer content) were mixed 30 minutes. The filler was introduced into the thus prepared composition and mixing was continued until a homogeneous form was obtained. The composition was then left to stand for 2 h for degassing. The adhesive composition prepared in this way was coated on a polyester film with a thickness of 36 µm using an automatic coating machine developed in the Laboratory of Adhesives and Self-Adhesive Materials of the West Pomeranian University of Technology in Szczecin in order to obtain the same thickness of the adhesive layer over the entire surface. The coated film was crosslinked in a drying channel at 120°C by 11 min. The ready-made adhesive tape was protected with polyester film.

Preparation of granite dust

Granite dust is prepared in two ways:

I. Granite dust/H_2O - The sample was washed with distilled water for 4 hours at 60 °C. 100 ml of distilled water per 50 g of sample were used. Everything was placed in an ultrasonic bath. The obtained samples were centrifuged (7,000 rpm) and dried at 85 °C for 24 hours.

II. Granite dust/CH_3OH - The sample was modified with a methanol solution for 4 hours at 60 °C. 100 ml of methanol per 50 g of sample was used for the modification. Everything was placed in an ultrasonic bath. The obtained samples were centrifuged (7,000 rpm) and dried at 85 °C for 24 hours. The fillers thus obtained were sieved to obtain a homogeneous dust which was added to the silicone resin.

Methods

Pot life is defined as the amount of time it takes for an initial mixed viscosity to double, or quadruple for lower viscosity products. It is timing starts from the moment when the product is mixed, and is measured at room temperature (23 °C).[12]

The value of shrinkage was measured using the well-known cross-method. The PVC or PET film was coated with a sample Si-PSA layer and cross-linked. Then it was applied on a degreased metal plate and two cuts were made at the right angle. The width of the cuts was measured. The measurements were taken at given time intervals, up to 42 days, at a measuring temperature of 70°C. The shrinkage is the percentage value of the ratios of width of the cuts. The shrinkage greater than 0.5% is not allowed.[13–14]

According to international standards (Association des Fabricants Europeens de Rybans Auto-Adhesifs and Fédération Internationale des Fabricants et Transformateurs d'adhesifs et thermocollants sur papiers et autres support) respectively AFERA 4001, FTM 8 and AFERA 4015 the adhesion, cohesion and tack was measurements.

Results and discussion

The preliminary pot-life tests for samples with the lowest filler content showed its gelation after 7 days from its receipt (Table 1). The situation confirms the analogous effect of granite powder on the properties of the non-cross-linked adhesive composition for silicone pressure-sensitive adhesives modified with silicon fillers such as kaolin or montmorillonite.[6–7,13] The rapid increase in viscosity of the stored composition prevents it from being too quickly prepared before coating.

The obtained composition exhibit a good useful properties (Table 2-3). In compared to the sample without fillers the addition of silicone fillers caused an initial increased useful value properties as adhesion or tack and then decline in their value. It may be caused by forcing a more compact structure of the adhesive film with small additions of filler (wt.% 0.1 - 0.5). Modification of silicone pressure-sensitive adhesives does not affect the cohesion of the adhesive film at room temperature (the cohesion value is maintained at a level of >72h). On the other hand, in the case of increased temperature, an addition of more than 0.5% by weight. shows the drastic shift of the adhesive-cohesive balance towards cohesion at the expense of adhesion, so that the samples did not withstand even 10 hours under load. It proves that the filler content is too high in the adhesive resin.

Table 1 Pot life of prepared silicone pressure-sensitive adhesives composition

Filler	Filler content [wt.%]	Viscosity [Pa*s]						
		1 day	2 day	3 day	4 day	5 day	6 day	7 day
Granite dust/H_20	0.1	16.7	17.4	21	25.1	32	37,5	gel
	0.5	21.2	21.8	24.5	30	43,5	44.2	gel
Granite dust/CH_30H	0.1	25.8	28.4	31.8	37.5	46	52	gel
	0.5	25.5	27.9	30.8	35.4	47	54	gel

Table 2 Influence of granite dust on cohesion in room and elevated temperature of Si-PSA prepared tape

Filler content [wt.%]	Cohesion			
	in 20°C		in 70°C [h]	
	Granite dust/H20	Granite dust/CH_30H	Granite dust/H_20	Granite dust/CH_30H
0	> 72	> 72	> 72	> 72
0.1	> 72	> 72	> 72	> 72
0.5	> 72	> 72	> 72	> 72
1	> 72	> 72	4.5	10.8
3	> 72	> 72	2.7	1.5
5	> 72	> 72	1.3	1.4
7	> 72	> 72	1.2	1.1

Table 3 Influence of granite dust on adhesion and tack of Si-PSA prepared tape

Filler content [wt. %]	Adhesion [N/25mm]		Tack [N]	
	Granite dust/H_20	Granite dust/CH_30H	Granite dust/H_20	Granite dust/CH_30H
0	8.67	8.67	13	13
0.1	10.65	13.75	13	15.55
0.5	12.55	14.15	12.7	13.25
1	7.93	8.27	11.25	11.25
3	7.69	7.95	10.15	10.48
5	7.48	7.8	8.88	9.25
7	7.04	7.31	8.47	8.99

Figure 1–2 show the change in shrinkage of cross-linked silicone-based pressure-sensitive adhesives depending on the filler additive. Along with the addition of the spreader, the shrinkage of the tested samples was reduced, which confirms the improvement of the cohesive properties of the adhesive films.[6–7] Moreover, washing in methanol allowed to increase the affinity of the filler to the polymer matrix, which can be seen in the diagram, where for the same granite dust fillings, the material modified with methanol showed a higher resistance to shrinkage.

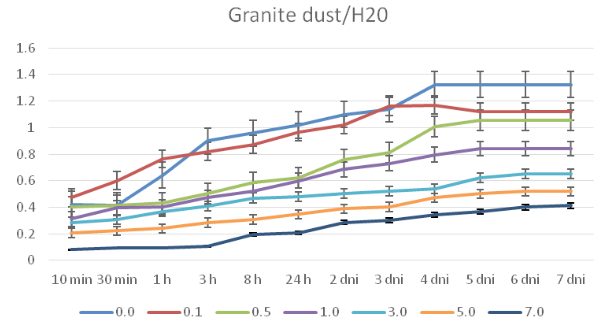

Figure 1 Influence of granite dust/H_2O on adhesion and tack of Si-PSA prepared tape.

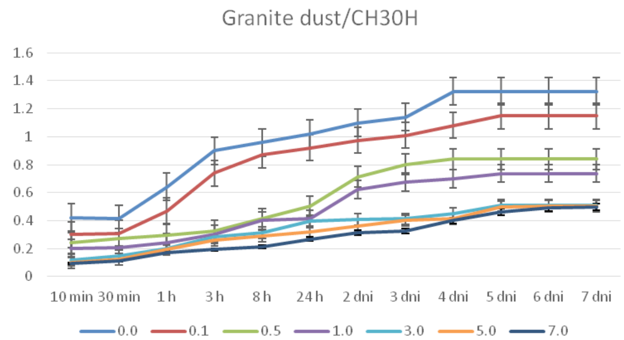

Figure 2 Influence of granite dust/CH_3OH on adhesion and tack of Si-PSA prepared tape.

Conclusion

Effect of addition cleaned granite dust on selected silicone pressure-sensitive adhesives was presented. The tested composition based on a prepared composition exhibit decreased of useful properties such as adhesion, tack, cohesion at elevated temperature. Properties like cohesion in room temperature has been kept to a high standard.

The presented research showed no positive interaction between the polymer matrix and the granite dust. The obtained results confirm the behavior of the filler at the level of an inorganic additive, which increases the functional properties of self-adhesive materials. The resulting tapes did not show any application potential for a while.

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