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Determining the wetting properties of mica with LPR-90 equipment

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Wetting and wetting rate measurements of a fine-grained mica were carried out by using the LPR-90 equipment. The mica samples had different kinds of surface treatment. It seemed that some significant differences could be observed as a function of the surface treatment agent and its concentration. According to the results, this equipment seems to be suitable for studying the success of surface treatments.

1. Introduction

The wetting of the reinforcements and fillers used in plastic composite technology with a matrix material under process conditions is a factor of primary importance [1]. In the case of thermosetting plastic resins this is generally no problem, because the process starts with unpolymerized thermosetting resins whose viscosity is low enough to achieve easy wetting. Wetting is particularly important in the case of thermoplast matrices, and the main problems involved here are illustrated in Fig. 1. Fig. 1a shows a situation where a matrix with a high viscosity cannot wet all surface structures, and Fig. 1b shows a situation where a thermoplast matrix cannot penetrate between the reinforcement components. This situation may occur, for example, while impregnating continuous reinforcement fibres with a thermoplastic resin.

The surfaces of fillers used for compounding thermoplastics are almost always modified. The aims of this modification are usually to improve the wetting of the reinforcement with the matrix material and/or to improve the adhesion of the reinforcement to the matrix material [2]. In these cases it is very difficult or even impossible to determine the success of such surface modifications by direct analytic methods.

2. Experimental procedure

In the present study the surface properties were measured indirectly by measuring their wetting under different conditions using the LPR-90 (Liquid Penetration Rate) equipment. The principle of the LPR-90 is shown in Fig. 2. It measures the absorption rate of liquid absorbed in a certain volume of material. At the beginning of the measurement there are two connected liquid surfaces that are on the same level. During the test one surface is brought into contact with the powder sample to be measured through a glass sinter, which wets very rapidly. The wetting rate of this glass sinter is high enough not to slow down the wetting of the specimen. When the specimen starts to absorb liquid, the level of the free liquid starts to sink. The equipment is provided with an optical detector connected to a dispensing pump which keeps the free liquid level constant throughout the entire measurement. In this way the equipment measures the amount of liquid absorbed into the specimen as a function of time [3].

The function of the equipment is based on the Washburn equation, which describes the wetting properties of a pulverous substance as a function of wetting speed by measuring the speed at which the liquid penetrates the powder and wets it. In practice the wetting does not occur exactly according to the Washburn equation, and consequently the method yields only a relative value of the wetting properties [3, 4].

The results obtained by this method are thus not absolute values indicating the wetting rate but values that correlate directly with the wetting. The method is useful for comparing the wetting properties of different liquid–mineral powder combinations. The results follow the curve shown in Fig. 3 (the results can be recorded either by plotter or directly with a computer), and the results indicate directly the penetration rate of the liquid into the powder, and, if a constant powder amount is used, the total liquid amount that is absorbed in a certain volume can also be measured.

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2.1. Test materials and objectives

The test material used in this work was a flogopite mica (Kemira OY, Siilinjarvi, Finland). Mica is typically sheet-like, and in the grinding it exfoliates into thin sheets. It is known as one of the most effective mineral fillers to increase the stiffness of thermoplastic compounds.

The objective of this work was to test the applicability of the LPR-90 equipment to measuring the wetting properties of mineral powders as a function of the properties of the powder. The property that we were mainly interested in was the effect of surface treatment, which was examined specifically in the case of mica. As regards the surface treatment, the aim was to examine the influence of the surface treatment substances themselves as well as the influence of variations in their concentrations.

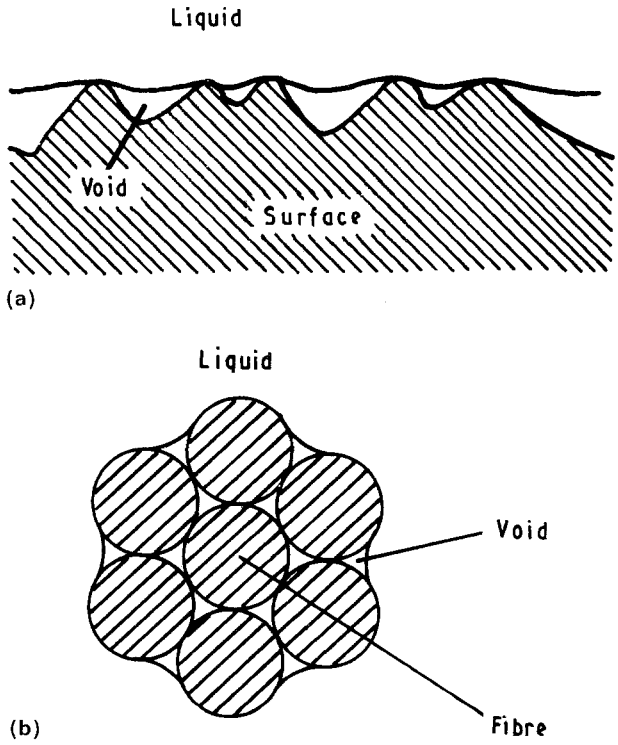

Figure 1 Poor wetting of (a) a solid surface or (b) a filler agglomerate.

3. Results

3.1. The wetting of mica

The wetting properties of mica were studied using a material with an average particle size of 40 μm. The analysis focused on the effects of the surface treatment of mica on the wetting properties.

First of all the wetting of mica without any surface treatment was measured, and these results were used as a reference for other results. The first actual test samples were the materials shown in Table I, which were used for testing the effect of the surface treatment material on the wetting. The surface treatment materials used here were three different silanes and one fatty acid. All of them are materials that have been found to improve the adhesion between mica and thermoplastic resins [5].

The second test series was the series shown in Table II, and its purpose was to find out the effect of the amount of silane used in the treatment on the wetting. The amount of surface treatment material was varied in the range 0.5–4.0 wt % of the amount of mica, because previously reported silane contents have generally been in this range, and the effects on mica–thermoplastic resin adhesion have appeared in this range. In theory even smaller silane contents

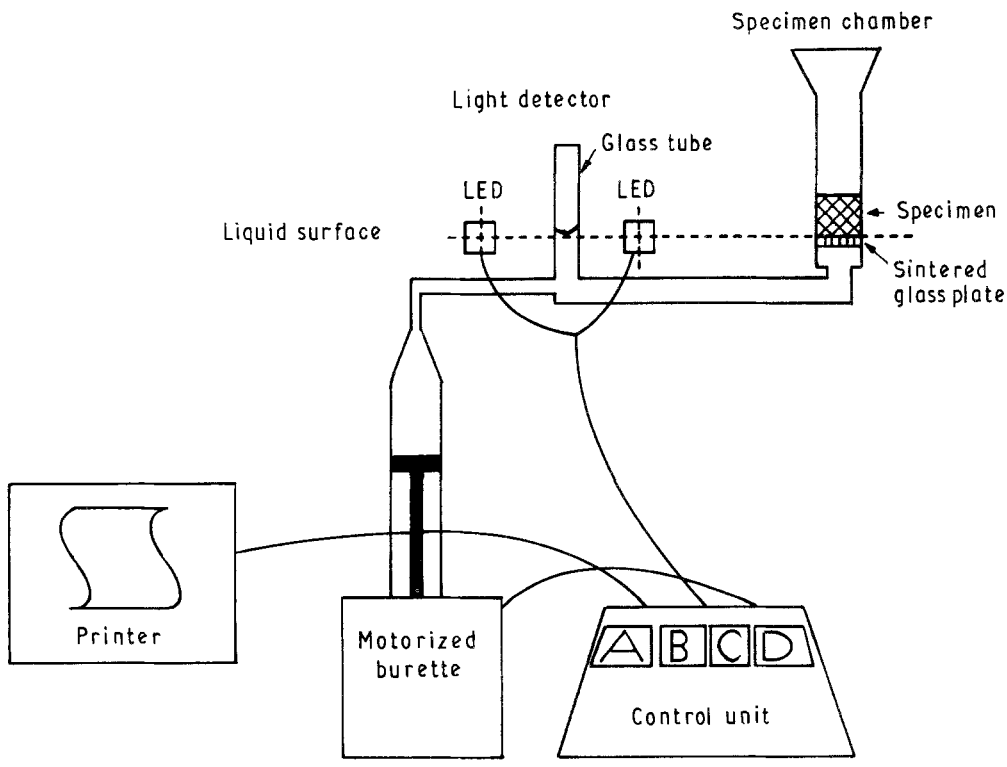

Figure 2 LPR-90 system and its operation principle [3].

TABLE I The micas used and their surface treatments

Material	Particle size (μm)	Surface treatment
Mica 40 A	40	None
Mica 40 B	40	None
Mica 40 S	40	Commercial material (silan A-1100 (Union Carbide), wet process)
Mica 40	40	0.5% silan A-1100 (UC)
Mica 40	40	0.5% silan A-174 (UC)
Mica 40	40	0.5% silan Z-6032 (UC)
Mica 40	40	0.5% polyacrylic acid

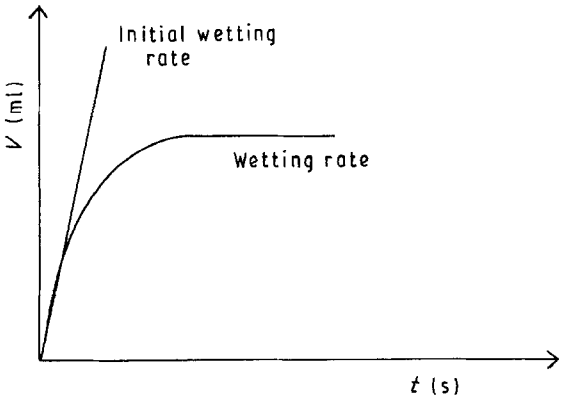

Figure 3 Wetting of a powder as a function of time measured by LPR-90 [3].

would suffice to cover the mica particles with a monomolecular silane layer. The higher silane contents are used because coating a fine-grained mineral powder evenly is very difficult. The test samples shown in Table II were coated using the so-called dry coating, where the silane is poured into the powder to be mixed mechanically. The silane spreads out on the particle surfaces when the particles touch. The test samples shown in Table I were coated with the so-called wet coating, where the mineral powder is dispersed in a dilute solution of the surface treatment agent and dried afterwards.

Fig. 4a–d show the wetting of mica treated with different surface treatment materials by water, acetone 1-propanol and n-heptane. The central properties for the wetting of the material with these liquids are shown in Table III. The results presented in Fig. 4 clearly show that the liquids wet the surface-treated mica specimens in different ways. The most distinctive effects are shown in the case of distilled water: pure mica, like silan A 1100, is hydrophilic and causes a rapid wetting. Other silanes, by contrast, make the mica more hydrophobic and slow down the wetting. Coating with a fatty acid has virtually no effect on the wetting properties. With other liquids the differences are smaller, but can still be observed.

Fig. 5a–c show the wetting rate for different liquids as a function of the amount of surface treatment agent. It is clearly evident that increased coating material concentration causes a distinct increase in the wetting rate with all liquids except acetone.

TABLE II Test series of micas with different amounts of surface modifier

Material	Particle size (μm)	Surface treatment
Mica 40	40	None
Mica 40	40	0.5% silan A-1100
Mica 40	40	1.0% silan A-1100
Mica 40	40	2.0% silan A-1100
Mica 40	40	4.0% silan A-1100

TABLE III Properties of wetting liquids used [7]

Liquid	Density ($kg\,m^{-3}$)	Surface tension ($mN\,m^{-2}$)
Water	1000	72.75
Acetone	792	23.70
1-propanol	804	23.78
n-heptane	680	20.30

The results also indicate that the surface treatment method has almost no effect on the wetting. This result is important because it means that different surface treatment methods are equally effective. This finding was confirmed by tests where mica was surface-treated with silan A 1100 (0.5 wt %) in both dry and wet processes.

3.2. Mica–polypropylene compounds

The types of mica and polypropylene under study were prepared as 30 wt % compounds. The specimens were made of these compounds by injection-moulding, and their mechanical strength properties were determined by tensile, flexural and impact tests. In addition, the dynamic properties of the compounds were characterized by DMTA analysis.

The main objective was to find out if the wetting rate values measured with the LPR equipment and the mechanical properties of the compound were correlated. Fig. 6 shows the flexural moduli and wetting rates in the case of mica specimens treated with different surface treatment materials.

In the light of Fig. 6 it is evident that the flexural strengths of surface-treated compounds are slightly higher than those of untreated compounds. A direct relationship between the mechanical properties of the compound and the wetting rates of fillers could not be observed.

It should also be noted here that surface treatments do not generally produce any notable improvements in mechanical properties in the case of pure polypropylene. This is clearly evident in Fig. 7, which shows the tensile and flexural strengths of compounds as a function of the surface treatment. It is also interesting to note that these surface treatment materials have virtually no visible effect on the strength. No ageing treatments were used on the materials, and all strengths were measured directly from moisture-stabilized test materials.

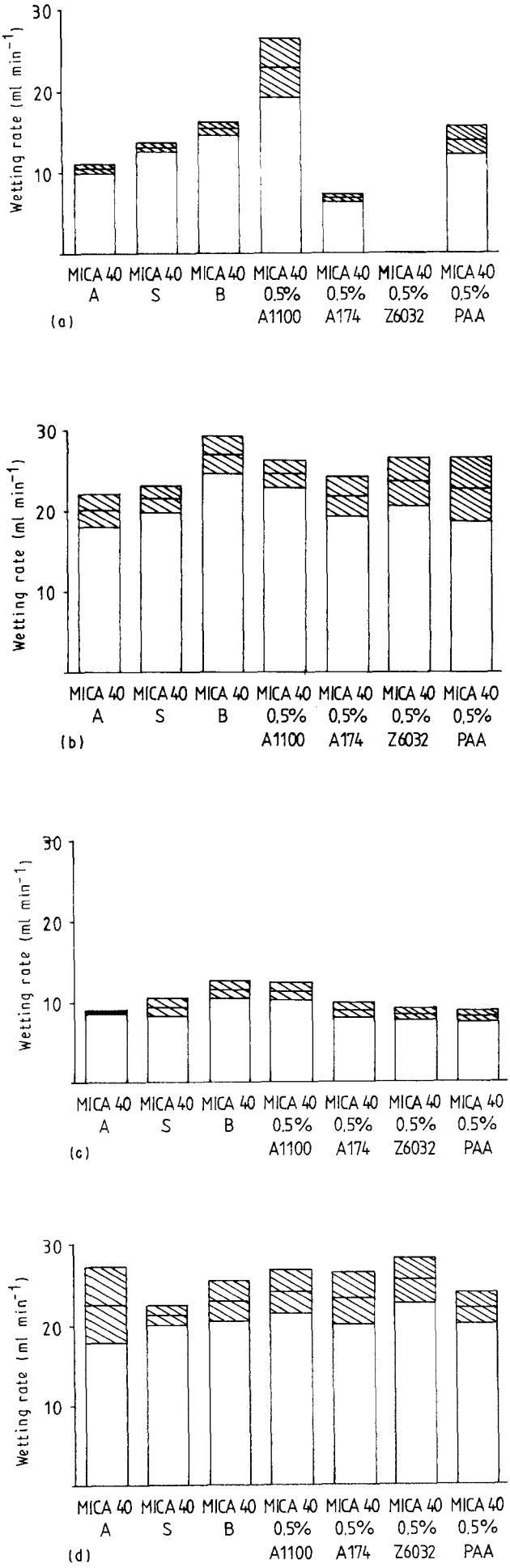

Figure 4 Wetting of mica with different liquids as a function of mica surface treatment (Table I): (a) distilled water, (b) acetone, (c) 1-propanol, (d) n-heptan [6].

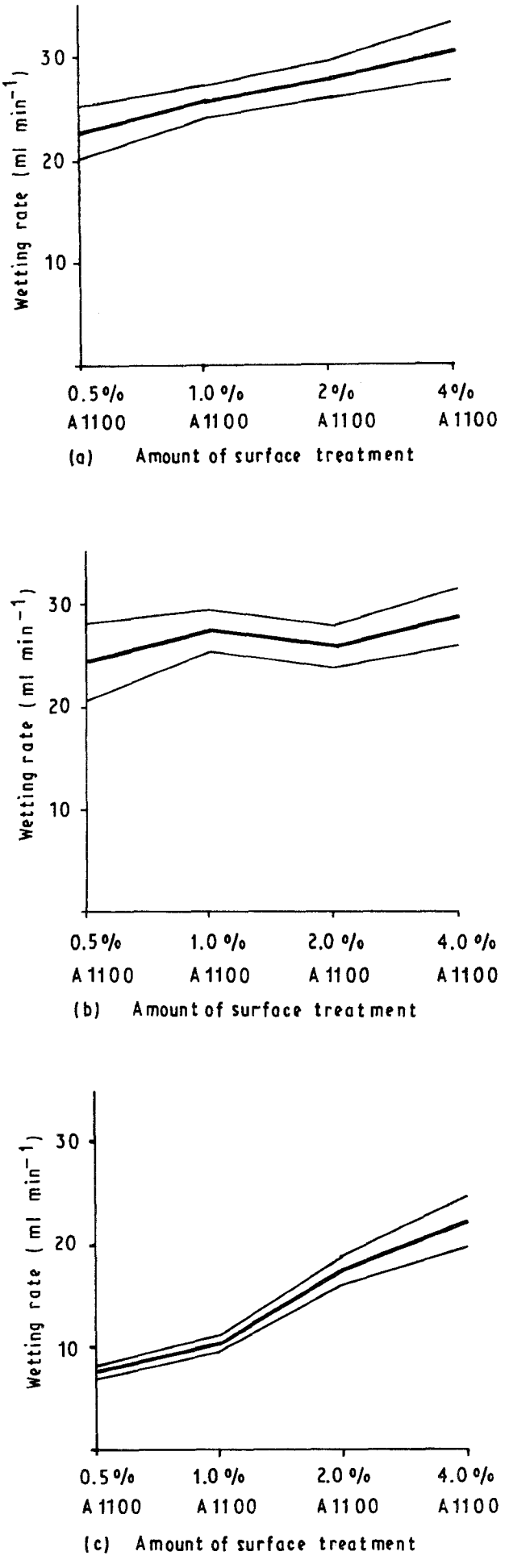

Figure 5 Wetting of surface-modified mica as a function of surface treatment amount with different liquids: (a) distilled water, (b) acetone, (c) 1-propanol [6].

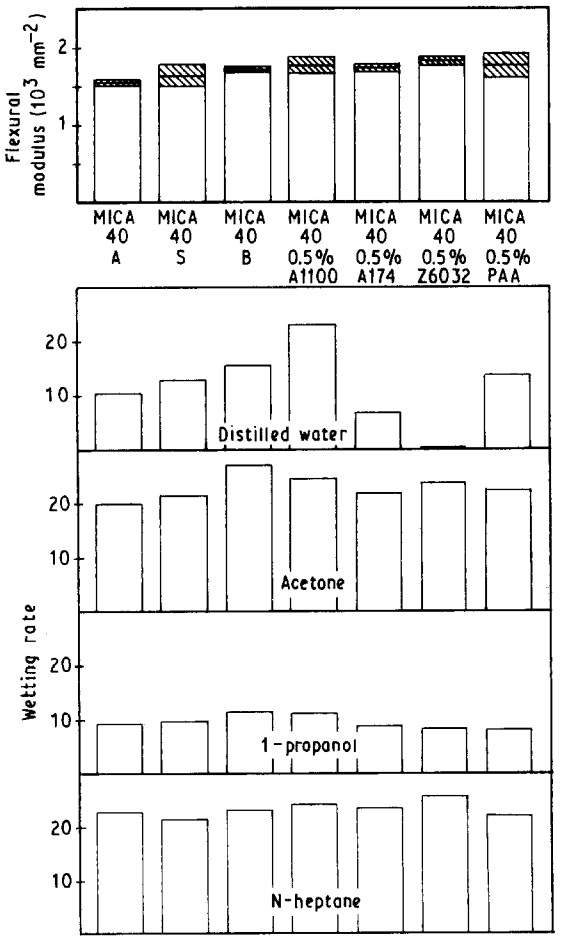

Figure 6 Flexural modulus of mica-filled polypropylene (30 wt % mica) and the wetting rate of mica with different liquids.

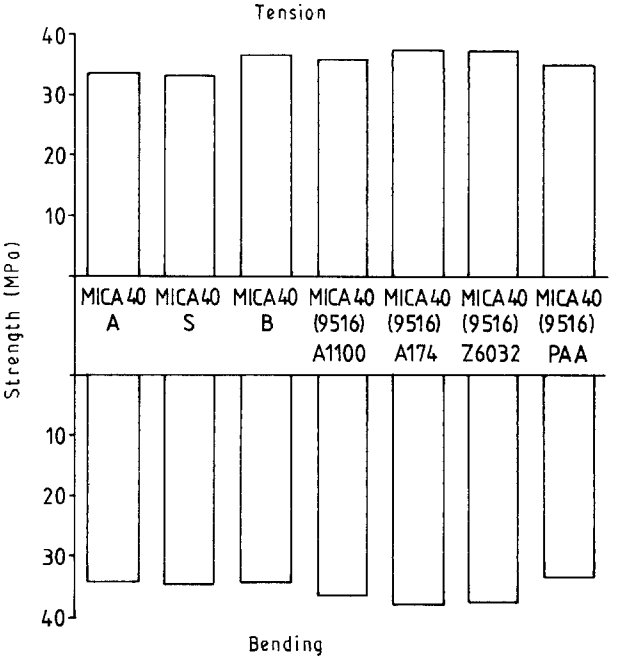

Figure 7 Tensile strength of mica-filled polypropylene (30 wt % mica) in the case of different surface treatments.

4. Conclusions

On the basis of the results the following main conclusions can be drawn. First, the LPR equipment has proved to be suitable for the wetting of various materials with different liquids. This is witnessed by the clear differences observed, for example, between similar mineral powders with different surface treatments.

The equipment is suitable for analysing the wetting by different liquids of such materials where the penetration of the liquid into the material occurs by capillary suction. The selection of liquid needs careful consideration because the wetting and wetting rate is strongly dependent on the polar and non-polar components of the surface tension for the liquid, as well as the polarity of the mineral surface [8]. The minerals to be wetted include all possible porous systems, such as mineral powders and mineral wools. In the light of the preliminary tests it seems that the equipment works rather well with many other materials as well, such as paper and cellulose materials when the wetting and wetting rate as such are of interest. The most suitable liquids for this method are liquids with a low viscosity. If the viscosity of the liquid is too high, it can block the channels between the powder particles to be tested.

As far as mineral fillers are concerned, the equipment is well suited for measuring, for example, the type and amount of surface treatment material. The results show very clearly the dependency of wetting between materials with different kinds of surface treatment and the amount of surface treatment agent. For this reason the LPR system is suitable to study the success of surface treatment. The results and differences have been verified with other methods and it is obvious that with a suitable selection of a series of liquids it would be possible to find a correlation between the wetting properties of the liquids and the matrix resins.

The question of the relationship between wetting and adhesion was left unanswered. It is very probable that wetting, adhesion and mechanical properties of the composites are not directly correlated at the interfaces of mineral-filled thermoplasts. It can be assumed that a good wetting is a prerequisite of adhesion, but it will not necessarily lead to strong bonds at the interfaces. The creation of strong bonds requires that the polymer and the mineral react together or through a mediating agent to create strong and stable bonds.

5. References

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