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QUANTITATIVE MODEL FOR SILICON-ACRYLATE MIXTURES

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Abstract: *Protection of surface layers of building materials are very important in construction industry. Plasters, which are used as a cover layer of load bearing structure, must resist to the adverse natural conditions, corrosion or mechanical damage. As the surface treatment there are used coatings which must require material and physical characteristics and help to resist to the material degradation. This article is focused on silicon-acrylate coatings. Silicons (polysiloxanes) are very resistant and expensive. Acrylates are cheaper and less resistant than polsiloxanes. The coating samples were measured in the ATR module in the infrared spectrometer. The advantage of this method is that the sample doesn´t need to be modified and the measurement is quite cheap. As a result, the mathematical model for quantitative analysis was created – the quadratic dependence of carbonyl peak area on the concentration of acrylate. Indirectly, the polysiloxane concentration can be calculated. In the reality the calculation of polysiloxane concentration can be the problem because of additives. The conditions of the measurement were specified and the statistical deviation was determined to be 4.6 %.*

Keywords: Silicon-acrylate mixtures, Quantitative model, Coating, Plasters.

1. Introduction

The surface layers of building materials are exposed to the adverse conditions in the surroundings (Pernicova, 2015). Therefore, it is absolutely needful that the material is resistant to the degradation and requires material and physical characteristics (Hodul, 2022). Currently, there are several options for protecting building materials. One of the often-used surface treatments are coating materials, that are applied to exposed surfaces (Hodul, 2022), however in some cases we also encounter internal coatings (Ticha, 2020). Load-Bearing materials such as concrete or masonry are covered with a protective layer - plaster. The plasters are also usually provided with a surface treatment. The combination of the matter of the plasters and the coatings is essential. Such modified plasters have to resist temperature changes, rain, microorganism growth, chlorides, air pollution, corrosion or mechanical damage (Pernicova, 2007).

The coatings that can be used in the construction industry are polysiloxanes, acrylates, epoxides and polyurethanes. Nowadays, there are increasingly used mixed coating materials because of improving physico-chemical properties. The polysiloxanes are polymers which are relatively constant in physico-chemical properties in a wide range of temperatures. Polysiloxanes aren´t reactive, are resistant to corrosion and microorganism. However, they are expensive. It is the reason why they are used in the mixture with acrylates which are cheaper. Acrylates are also less resistant than polysiloxanes. The problem of these mixtures is obvious – it cannot be easily determined the chemical composition of the mixtures – quantity of polysiloxanes and acrylates.

In the case of surface coatings, its functional properties, quality of application using the laser microscope system (Reiterman, 2013) and also its chemical composition are primarily determined. The aim of this article is to design the model for quantitative analysis of polysiloxanes and acrylates with the infrared spectroscopy.

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2. Methods and Materials

The analytical methods for determination of chemical composition are e.g atomic absorption or atomic emission spectroscopy, liquid or gas chromatography or mass spectrometry. The methods of the sample treatment are different – samples can be solid, liquid or gaseous. The coating sample is solid. The HPLC method (high pressure liquid chromatography) is a method that is very accurate for quantitative analysis of polysiloxanes, polyurethanes and other sunstances present in coating mixtures but it is also very expensive method. The solid sample must be transferred into the liquid form. The cleaning of the colony is time-consuming. Moreover, it is not easy to use it in the industry. The indisputable advantages of the infrared spectroscopy are easy measurement technique, the sample can be measured as it is, it can be also used in the industry. The main disadvantages of these technique are lower detection limit for organic substances. This method is often used even by historians or employees of the cultural heritage office (Sladka, 2020), because the requirements for the material composition (especially for plasters) are very strict and specific (Vejmelkova, 2009).

Infrared spectroscopy is method based on the quantum mechanics where the photon is absorbed by the matter and then emitted with different frequency (Panowicz, 2011). The experimental set-up of infrared spectrometer is shown in the figure 1. There is photon source, mirrors, sample space and detector. At the beam splitter there is division of one photon and then interference of two photons with the phase difference. This interfered photon with higher intensity is absorbed by the sample and then the emitted photon is detected by the detector and the signal is converted into the spectrum by the Fourier-transformation.

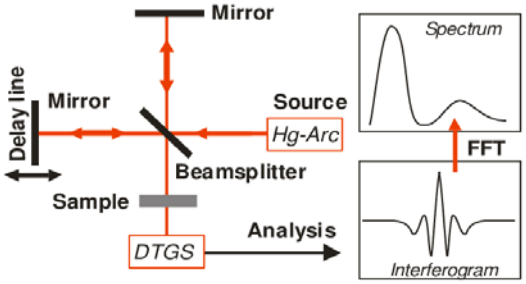

Fig. 1: Experimental set-up of infrared spectrometer (Dexheimer, 2008)

The infrared spectroscopy can determine the functional groups present in the sample. Supposed that the sample is mixture of polysiloxanes and acrylates. Acrylates are esters of acrylic acid and its derivates. The formula of the ester is CH_2=CH-COO-R, where R is alkyl group. It contains following bonds: C-C, C=C, C-O-C, carbonyl group C=O and vinyl group CH_2=CH-. Polysiloxane´s formula is $[R_2SiO]_n$, where R is organic substituent. This polymer chain contains only bonds between Si-O and bonds between Si-C. It is seen from the formulas and from the figures 2 and 3 that the spectrum of polysiloxanes is easier than the spectrum of acrylates. The quantitative analysis from the infrared spectrum can only be determined from the separate peaks without the overlay. In the figure 2 there is the spectrum of acrylate where only two separate peaks are – peak at 1730 cm^{-1} and at 880 cm^{-1}. In the polysiloxane´s spectrum there is only one peak without the overlay – peak at 1280 cm^{-1}. However, there are more peaks at this frequency in the acrylate spectrum. The presence of polysiloxane can only be detected indirectly from the presence of acrylate and it can be more inaccurate.

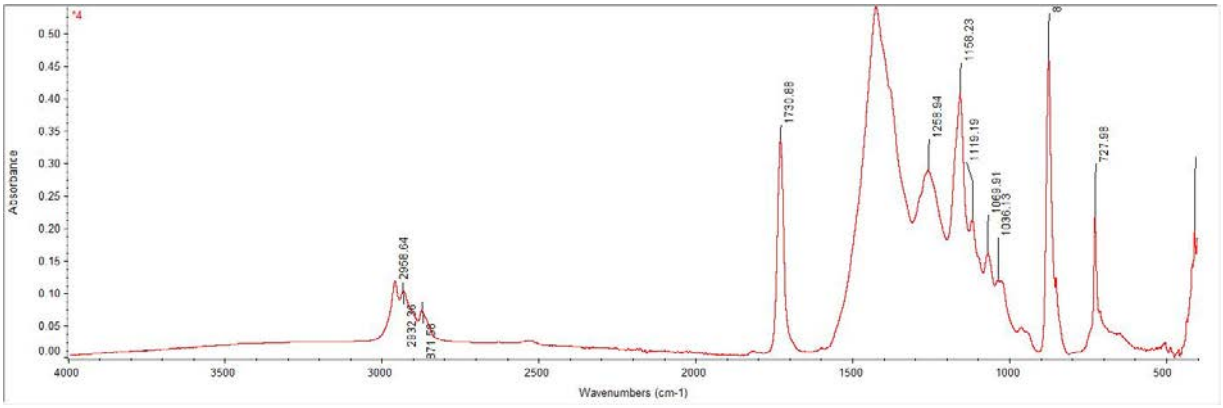

Fig. 2: Spectrum of acrylate.

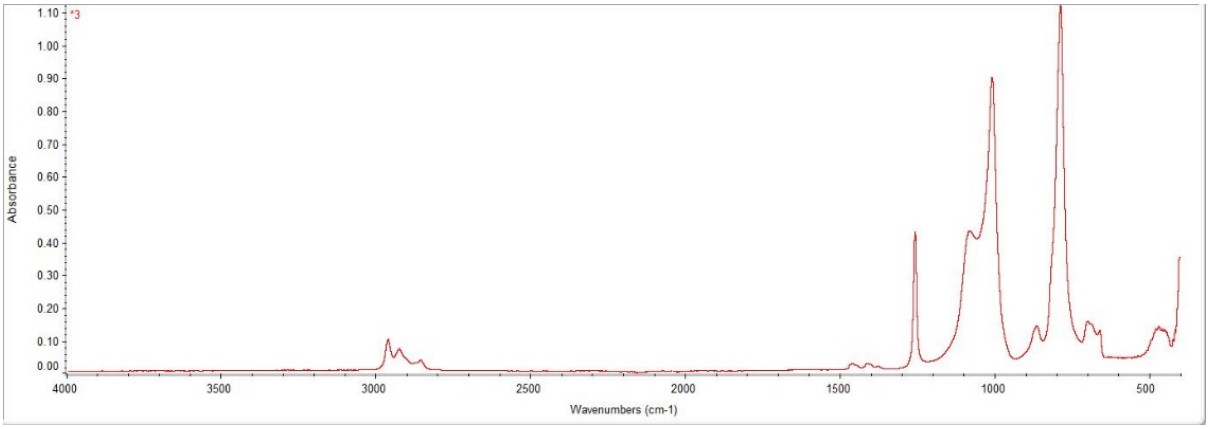

Fig. 3: Spectrum of polysiloxane.

The biggest difference in these spectra is the presence of the carbonyl peak at 1730 cm^{-1}. In polysiloxane´s spectrum this peak is missing. The intensity of the peak depends on the concentration of acrylate. This peak was chosen for analysis and for calibration. The spectra were measured by the ATR module of infrared microscope with diamond crystal. The samples were prepared as the mixtures of polysiloxane and acrylate on the laboratory glass and they weren´t further modified. The spectra were evaluated in the OMNIC software. The analysis was performed at the different parts of the sample and the results are the average values from the several measurements. The spectra were baseline corrected and the measured parameter was the peak area at the frequencies 1675 – 1775 cm^{-1}.

2.1. Results and discussion

In the figure 4 there is the model, which the chemical composition of silicon-acrylate mixture is determined from. The quadratic equation is

$$A = 2.3424w^2 - 0.0983w + 0.0097, \quad (1)$$

where A is the peak area and w is the mass fraction of acrylate in percent. The reliability coefficient is 95.5 %.

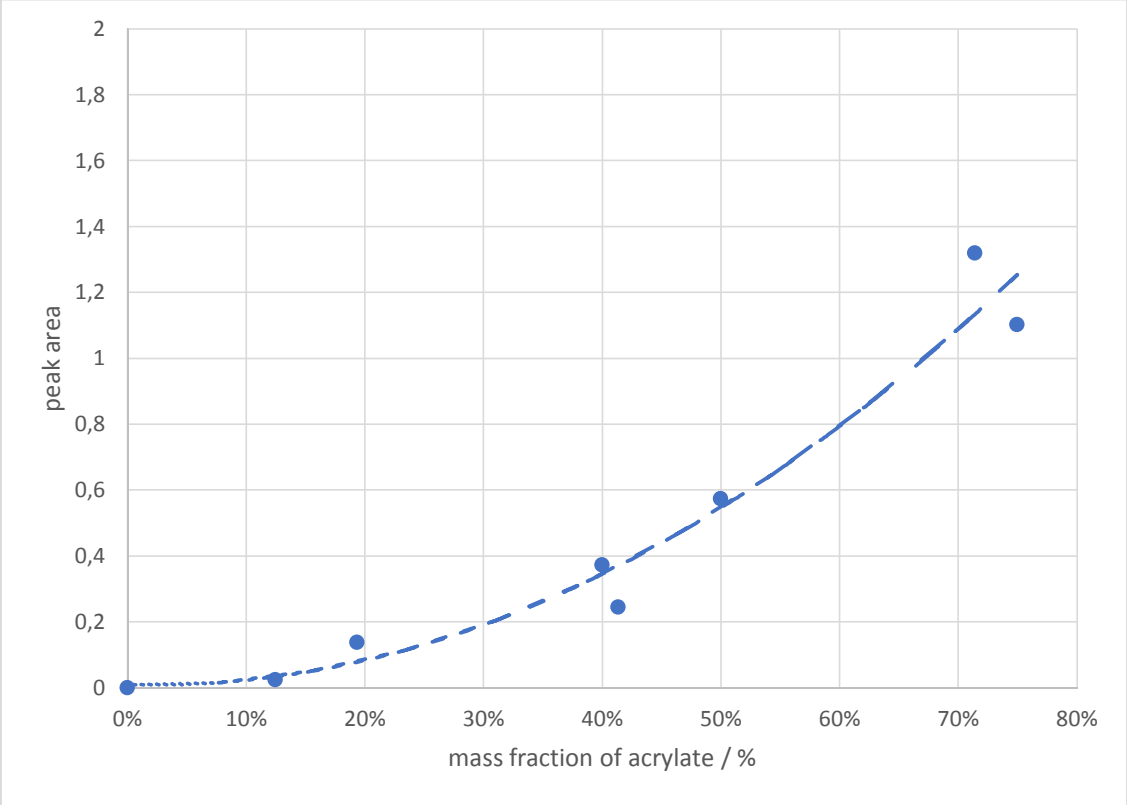

Fig. 4: The model for quantitative analysis of silicon-acrylate mixtures.

My sample for the model reliability verification was silicon-acrylate sample with the following mass fractions:

$$w_{silicon} = 0.56 \tag{2}$$

$$w_{acrylate} = 0.44 \tag{3}$$

The sample was measured and analyzed in the ATR module in the infrared spectrometer with the same parameters as the calibration samples. The peak area was A = 0.335 and the mass fractions of silicon and acrylate were calculated from the model above. The theoretical mass fractions were:

$$w_{sil,theo} = 0.61 \tag{4}$$

$$w_{acr,theo} = 0.39 \tag{5}$$

The calculated measurement deviation was 4.6 %. The deviation is larger with lower acrylate concentrations. The difference between intensity of carbonyl peak is small at low acrylate concentrations. Therefore, the statistical deviation is larger in these experiments.

3. Conclusions

Within this article the mathematical model for determination of polysiloxane and acrylate was prepared. The equation for calculations was $A = 2.3424w^2 - 0.0983w + 0.0097$, where A is the area of the carbonyl peak and w is the acrylate mass fraction in percent. The value of reliability R^2 is 95.5 %. The statistical deviation is larger with the lower concentration of acrylate – for the sample with $w_{acrylate} = 0.44$, the measurement deviation is 4.6 %. The concentration of polysiloxane can only be determined indirectly and it strongly depends on the additives in the coating materials.

Acknowledgement

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