Strojnícky časopis – *Journal of* MECHANICAL ENGINEERING,
VOL 75 (2025), NO 1, 147 – 154

METHODS OF WATER REMOVAL FROM LIQUID EPOXY RESIN

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Abstract: The aim of the article is to show the possibilities of releasing water from liquid epoxy resin. It is important to perform this procedure with the resin that is to be used for the lamination process. Water-contaminated resin negatively affects the polymerization process of epoxy resin and has an adverse impact on the mechanical properties of the resulting laminate. This article describes two methods of releasing water from resin. The water content in the resin was measured spectrometrically (NIR spectrometry)

KEYWORDS: epoxy resin, polymer composite, glass/epoxy laminate, NIR spectrometry

1 Introduction

Epoxy resin is one of two components of polymer composite materials [1][2][10]. Epoxy resin is a component of a composite material that, after the polymerization process, forms the so-called polymer matrix of the resulting composite. During the curing process, the epoxy resin changes from a liquid state to a solid state, forming a three-dimensional network structure of polymer matrix.

It is important that only two components enter the curing process – the epoxy group (oxirane circle) and the amine hardener. In this case, the formation of the 3D structure of the polymer matrix will not be disturbed by anything (provided that the temperature, type of resin and hardener and their mass ratio are maintained). In practice, it often happens that a third component also participates in the reaction – water, which is introduced into the reaction by the resin. Water enters the resin most often as air humidity, which the resin binds to itself. One of the main disadvantages of epoxy resins is their relatively high ability to absorb water [3][4]. Water in the resin worsens its thermomechanical properties and adhesion, and causes chemical degradation of the net polymer structure.

It has been proven that the water content in epoxy resin adversely affects the mechanical properties of the resulting laminate, in the production of which such water-contaminated resin was used [5][6].

It is therefore important, before the lamination process, to remove water from the epoxy resin.

For this purpose, we tried two methods:

- heating the resin
- drying the resin with silicagel

For our laboratory experiments, epoxy resin LH 287 was used.

DOI: 10.2478/scjme-2025-0015, Print ISSN 0039-2472, On-line ISSN 2450-5471

2 Water release from epoxy resin by heating

One way to remove water from epoxy resin is to heat it. By heating the resin above the boiling point of water and simultaneously stirring it vigorously, it is possible to remove water in a relatively short time. It is important not to exceed the boiling point of the resin itself, which ranges from 150-350 °C, depending on the type of resin.

At the beginning of the experiment, the anhydrous resin (absorbance value 0.85) was contaminated by adding 5 wt. % of water. The sample was then homogenized by stirring to ensure an even distribution of the added volume of water in the volume of the resin.

Fig. 1 Water contaminated resin (left) and pure epoxy resin (right)

The resin was then heated to 120 °C (at this point the resin absorbance value was 2.2), and the resin was maintained at this temperature throughout the experiment. From this point on, the decrease in water content was monitored over a 60 min time frame using NIR spectrometry [7][8][9] – measuring the resin absorption spectrum in the NIR region. The absorbance value in the region ~1900 nm was measured in individual samples taken at defined time intervals from the heated resin.

Absorbancy (A) is given by equations

$$\log\left(\frac{\Phi_0}{\Phi}\right) = \varepsilon . c . d \qquad (1)$$

$$A = \varepsilon . c . d \qquad (2)$$

ε – molar absorption coefficient
c – concentration
d – thickness of the liquid layer

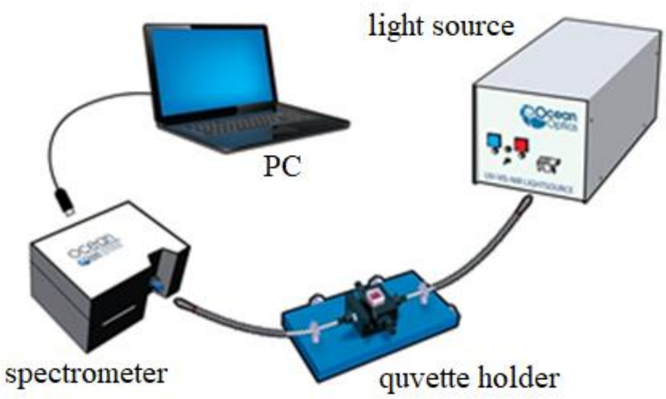

Fig. 2 NIR spectrometric system

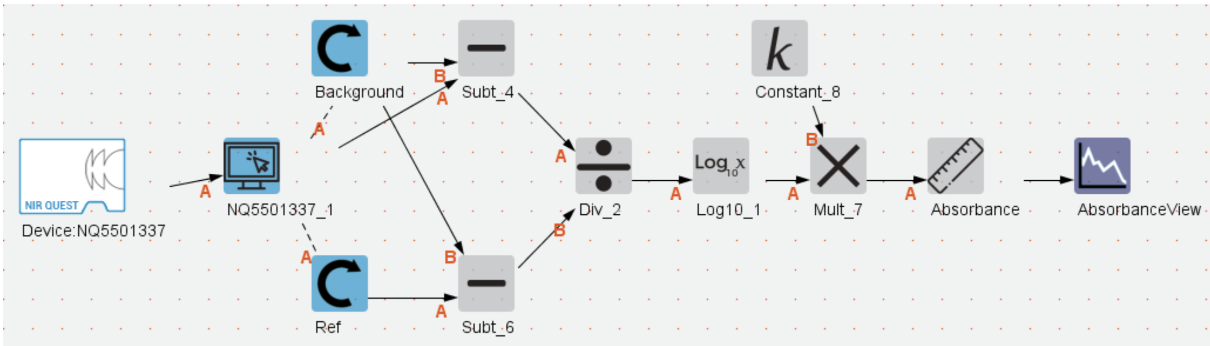

Fig. 3 Ocean View spectrometric computational scheme

Due to the signal sampling period of the Ocean Optics NIRQuest+2.5 spectrometer used, it was not possible to accurately measure the wavelength of 1900 nm (the region of light absorption by water). Therefore, the closest wavelength value (to the value of 1900 nm) that the spectrometer used allows to measure was chosen, 1901.938 nm.

The following graph (Fig. 4) shows the spectral profile of the water-contaminated resin at the beginning of the experiment (blue curve) and at the end of the experiment (red curve), when the water was practically removed from the resin. In the wavelength region ~1900 nm, there is a clearly visible peak representing the absorbance of the resin, which decreased during the experiment from a value of 2.2 (5 wt. % of water in the resin) to 0.85 (0 wt. % of water in the resin).

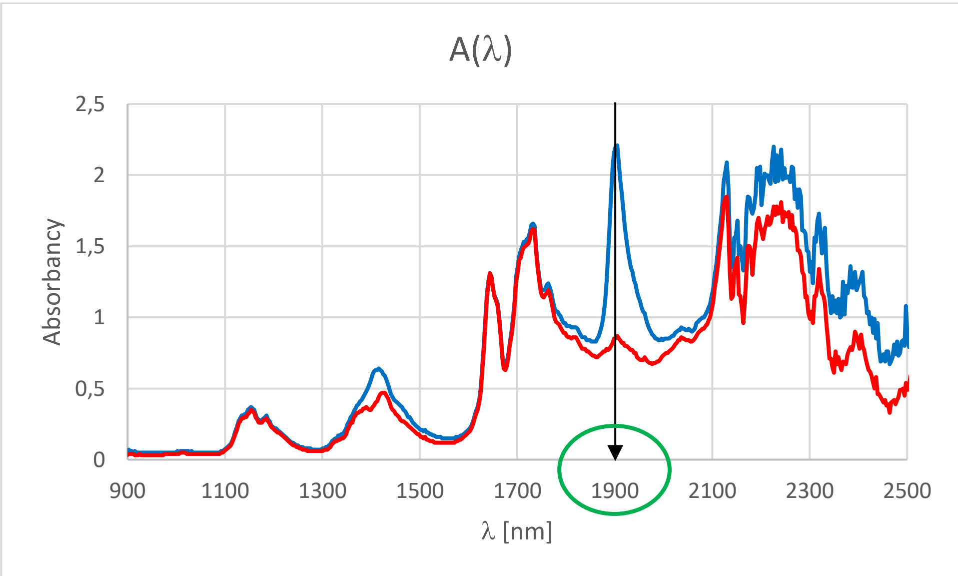

Fig. 4 Spectrometric profiles of water contaminated and pure epoxy resin

The gradual decrease in water content represented by the decrease in the peak in the region of ~1900 nm (green circle) is shown in a detailed view (wavelength range 1850-1950 nm) in Fig. 5. The complete course of the experiment in the form of a decrease in the absorbance value during heating of the resin is given in Tab. 1.

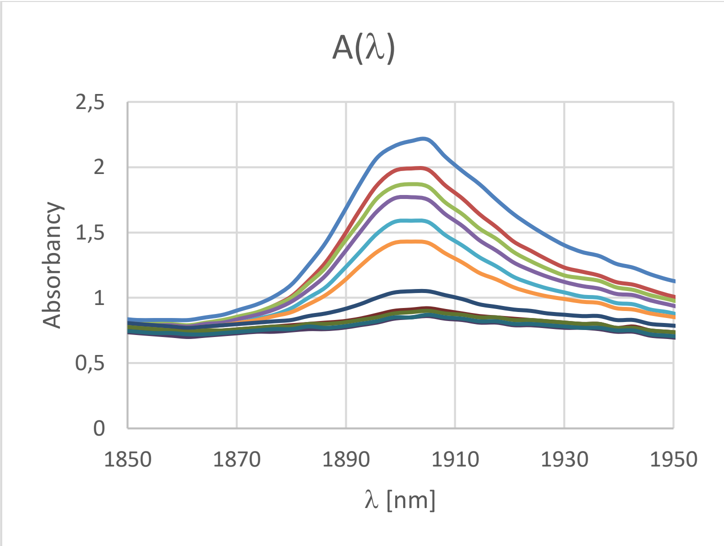

Fig. 5 Detail view of 1900 nm area

Tab. 1 Change of resin absorbance as a function of heating time

t [min]	0	1	2	4	7	10	20	30	40	50	60
A	2,2	1,99	1,87	1,77	1,59	1,43	1,05	0,91	0,89	0,85	0,85

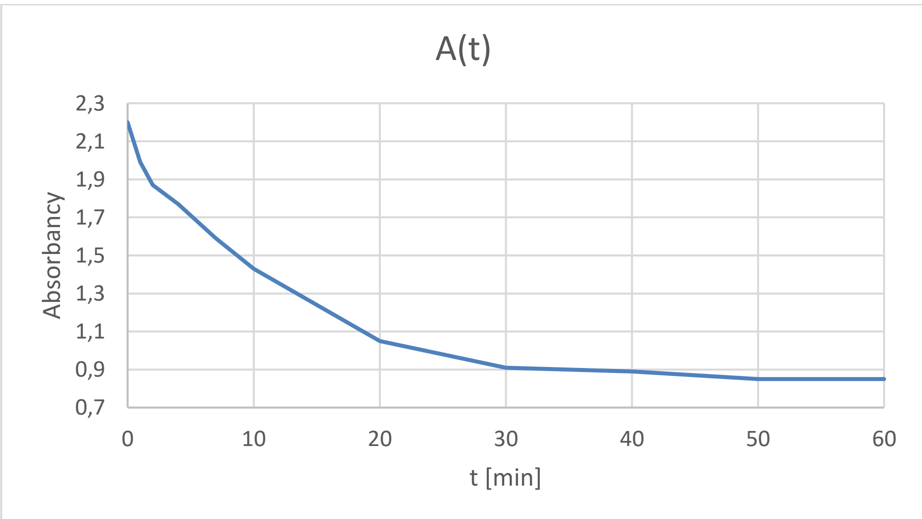

Fig. 6 Decrease of resin absorbance as a function of heating time

In Fig. 6 it can be seen that a significant reduction in water content can be achieved after just 20-30 min. of heating. From a practical point of view, continuous heating for 60 min. is sufficient to remove most of the water content from the resin.

3 Water release from epoxy resin by drying

Second method of water removal from epoxy resin is its drying with silicagel. The drying process took place in a hermetically sealed box, on the bottom of which a layer of silicagel was poured. Five beaker with water contaminated with resin were placed on this layer. The contamination level was in the range of 1-5 wt. %. The resin in the individual containers was stirred once a day, throughout the entire duration of the experiment.

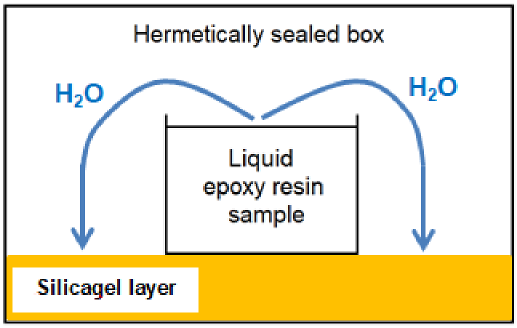

Fig. 7 Experiment setup of epoxy resin drying by silicagel

The drying process was monitored by measuring the resin absorbance in the region of ~1900 nm, in individual samples taken at defined time intervals from the dried resin. Since the process of drying epoxy resin with silicagel is much more lengthy (compared to the method of heating the resin), the samples were taken at intervals of days. The measured values are given in Tab. 2.

Tab. 2 Epoxy resindrying process

	Water content in epoxy resin (wt. %)				
	1%	2%	3%	4%	5%
Day	A	A	A	A	A
1	1,65	1,84	1,87	2,06	2,20
2	1,52	1,66	1,73	1,81	1,88
5	1,31	1,36	1,40	1,48	1,48
6	1,27	1,34	1,32	1,38	1,41
7	1,22	1,25	1,29	1,33	1,35
8	1,21	1,25	1,26	1,29	1,31
9	1,19	1,21	1,22	1,25	1,27
12	1,12	1,13	1,13	1,18	1,19
14	1,12	1,14	1,14	1,14	1,16
22	1,08	1,09	1,10	1,09	1,12
26	1,12	1,09	1,09	1,09	1,10
30	1,09	1,09	1,09	1,09	1,10
34	1,12	1,10	1,10	1,09	1,08
40	1,10	1,10	1,10	1,10	1,10

A graphical representation of Tab. 2 is shown in Fig. 8. This figure shows the time dependences of absorbance (A as a function of t) for individual contamination levels (1-5 wt. % of water).

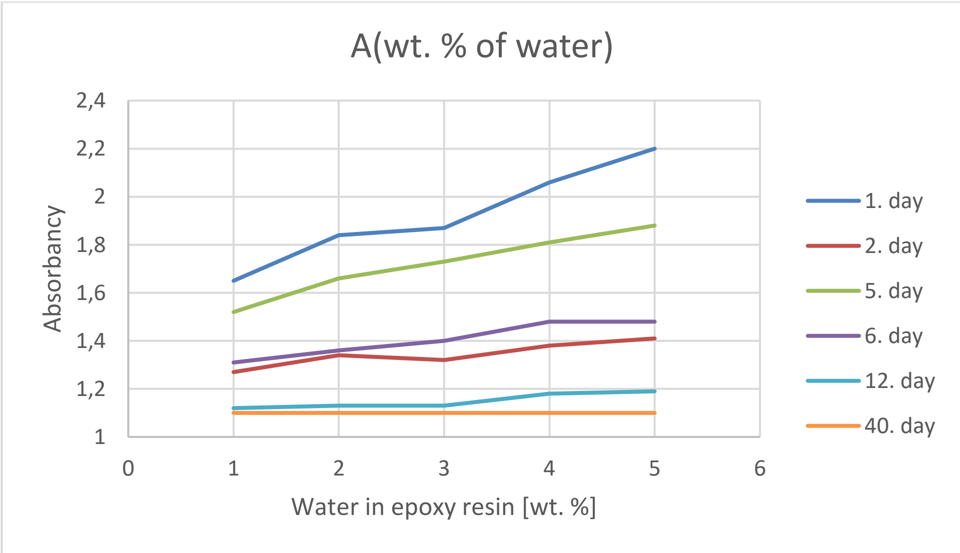

Fig. 8 Tab. 2 visualization of chosen rows (representative days)

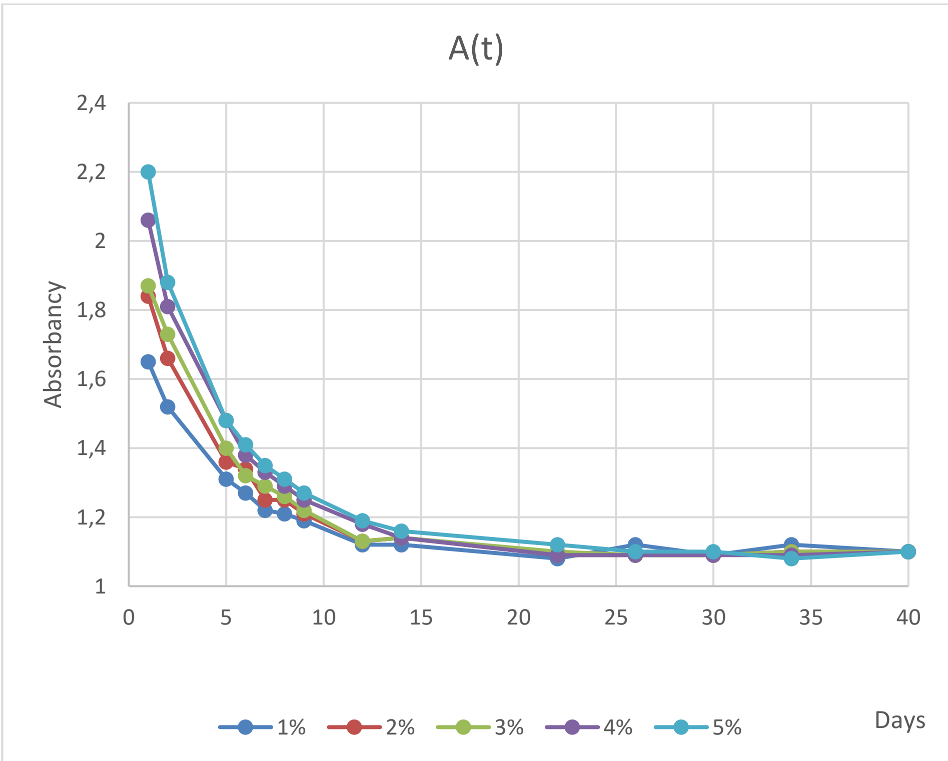

Fig. 9 Visualization of individual columns of the Tab. 2

The graphs (Fig. 8 and Fig. 9) show a higher time requirement of the drying process (approx. 22 days) and its lower efficiency (absorbance 1.1), compared to the method of heating the resin.

CONCLUSION

Using epoxy resin with the lowest possible water content is important for the correct course of its reaction with the hardener. The amount of water has a negative impact on the mechanical properties of the resulting polymer composite. In this article, we have described two methods of water removal from epoxy resin – heating the resin and drying the resin with silicagel.

By heating the resin to a temperature of ~120 °C, it is possible to achieve a significant reduction in water in the resin in 20-30 min. and its practically complete removal after 60 min. After 60 min. of heating, the absorbance value (0.85) was achieved, which is equal to anhydrous resin.

The second method – drying the resin with silicagel – is less effective. It takes longer and the reduction in water content in the resin is smaller. After 22 days of drying under controlled conditions, the absorbance was ~1.1, which is a worse result compared to heating. If an anhydrous resin is defined by an absorbance of 0.85 and a resin with a water content of 1 wt. %, by an absorbance of 1.65 (see Tab. 2), then the absorbance of 1.1 achieved by drying can be equated to approx. 0.5 wt. % water in the resin (taking into account the strongly simplifying assumption of a linear dependence of the absorbance on the water content, on the interval 0 to 1 wt. % water, or on the absorbance interval 0.85 to 1.65).

ACKNOWLEDGEMENT

This research was supported by the Slovak Research and Development Agency under Contract No. APVV-20-0428.

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