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ORIGINAL PAPER

THERMAL AND RADIATION STABILITY OF ALKYD BASED COATINGS USED AS INSULATORS IN THE ELECTRICAL ROTATING MACHINES

MARIUS BUMBAC[1], TRAIAN ZAHARESCU[2], CRISTINA MIHAELA NICOLESCU[3]*

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Abstract. *Alkyd resin coatings are applied, mostly, due to their versatility, proper stability, and good compatibility with other modifying agents like curing agents, antioxidants, and nanoparticles that allow manufacturing resins with improved properties. The proportions between mixture components as well as the control of curing process parameters influence the final features of the final product.*

In this study, three types of alkyd resins consisting of phthalic anhydride, pentaerythritol, benzoic acid, propylene glycol as well as three different type of vegetable oil, were investigated. The content of vegetable oil was the same. In all alkyd formulations, for the curing process, melamine resin was used and the resulted mixture was tested for thermal and radiation stability. Epoxy resin, acrylamide, and SiO_2 *and* Al_2O_3 *nanoparticles were used for the improvement of the material. The structural change of obtained polymers was investigated by IR spectroscopy with attenuated total reflection (ATR) and conclusions about their stability were drawn. The thermal stability of irradiated and non-irradiated samples was investigated by chemiluminescence in stationary air atmosphere, as well.*

Keywords: *alkyd resin, melamine, thermal stability, IR spectroscopy, flame retardant*

1. INTRODUCTION

Alkyd resin coatings have various applications such as chemical and mechanical protection of some parts of the industrial equipment with high usage rate [1]. Their role in the coatings production is very important for two main reasons: their versatile characteristics and low cost of the synthesis using natural raw [1-4]. However, there are some drawbacks of the alkyd resins, such as low alkali resistance, long curing time, weak surface adhesion and low hardness [5].

In the same time, alkyd resins have good compatibility with a large variety of resins (amino-resins [6], synthetic dyes [7], and silicon resins [8]). The incorporation of various types of resins causes the improvement of functional features. The combination of alkyd resin with melamine resin leads to a cured film having the flexibility of the alkyd component. Simultaneously, the obtained resins receive the strength and hardness of melamine resin [9-11].

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[1] Valahia University of Targoviste, Faculty of Sciences and Arts, 130082 Targoviste, Romania.

[2] National Institute for Research and Development in Electrical Engineering ICPE-CA, 030138 Bucharest, Romania.

[3] Valahia University of Targoviste, Institute of Multidisciplinary Research for Science and Technology, 130004 Targoviste, Romania. E-mail: cristina.nicolescu.profa@gmail.com.

*Corresponding author: cristina.nicolescu.profa@gmail.com

The curing process of resin is carried out by a complex series of transformations, and some of them are reversible. For instance, during this process, some bonds may be broken and reformed several times leading to significant and continuous changes in the cross-linked structure of polymer [11].

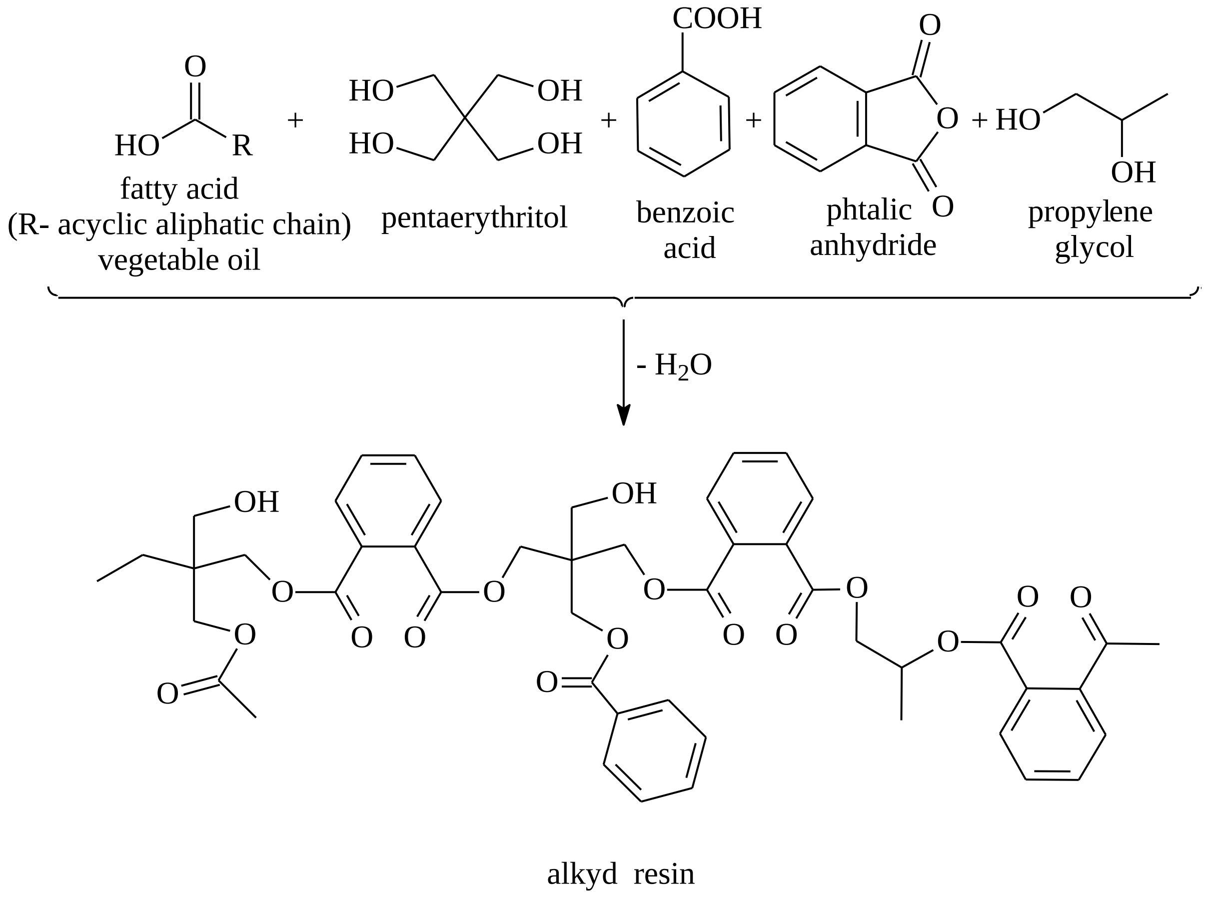


Figure 1. Possible crosslinking structure in alkyds.

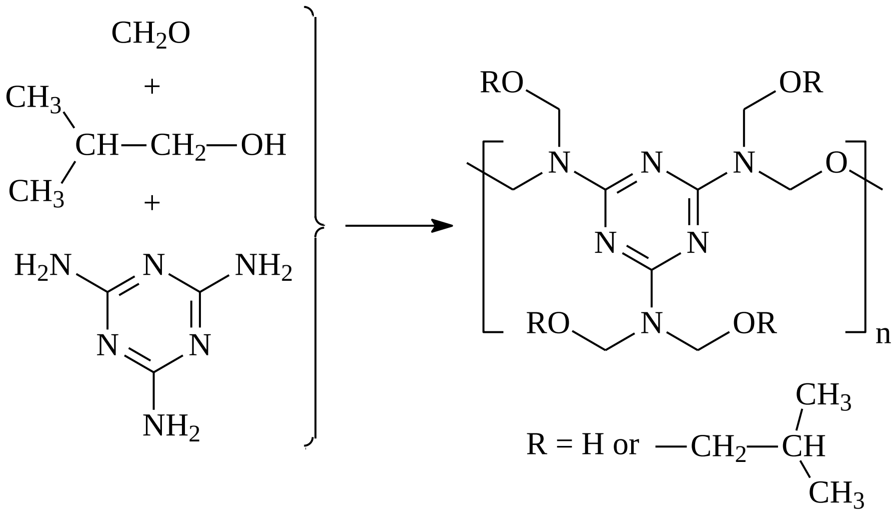


Figure 2. Structure of poly(melamine-*co*-formaldehyde) isobutylated resin.

Gan and Tan studied the curing process of alkyd resins based on palm oil and melamine by analyzing the IR spectrum modifications. They found that formation of methylene-ether bonds is the dominant reaction [12]. Amino resins may be also combined with various alkyd resins based oils such as sunflower oil, soybean, castor oil, tung or several mixtures of these. Alkyd resin contains carboxylic groups and free hydroxyl groups as well. This configuration shows two advantages: the first benefit is the presence of hydroxyl groups involved in the cross-linking process, and the second advantage consist of the catalyst role of the carboxylic groups along this process [4].

The increase in amino resin content would cause the increase of the hardness of cross-linked material, the improvement of resistance to chemical, thermal and to photo-oxidation [16-18]. On the other hand, an increased percentage of alkyd resin content, leads to a higher flexibility and adhesion properties in polymeric material [19].

Figure 3 shows a cross-linking way for polymer structures synthesized by mixing alkyd resin and melamine resin that were further heated at 150°C. Possible bonds in the final compound, which may be formed during the curing process, were suggested.

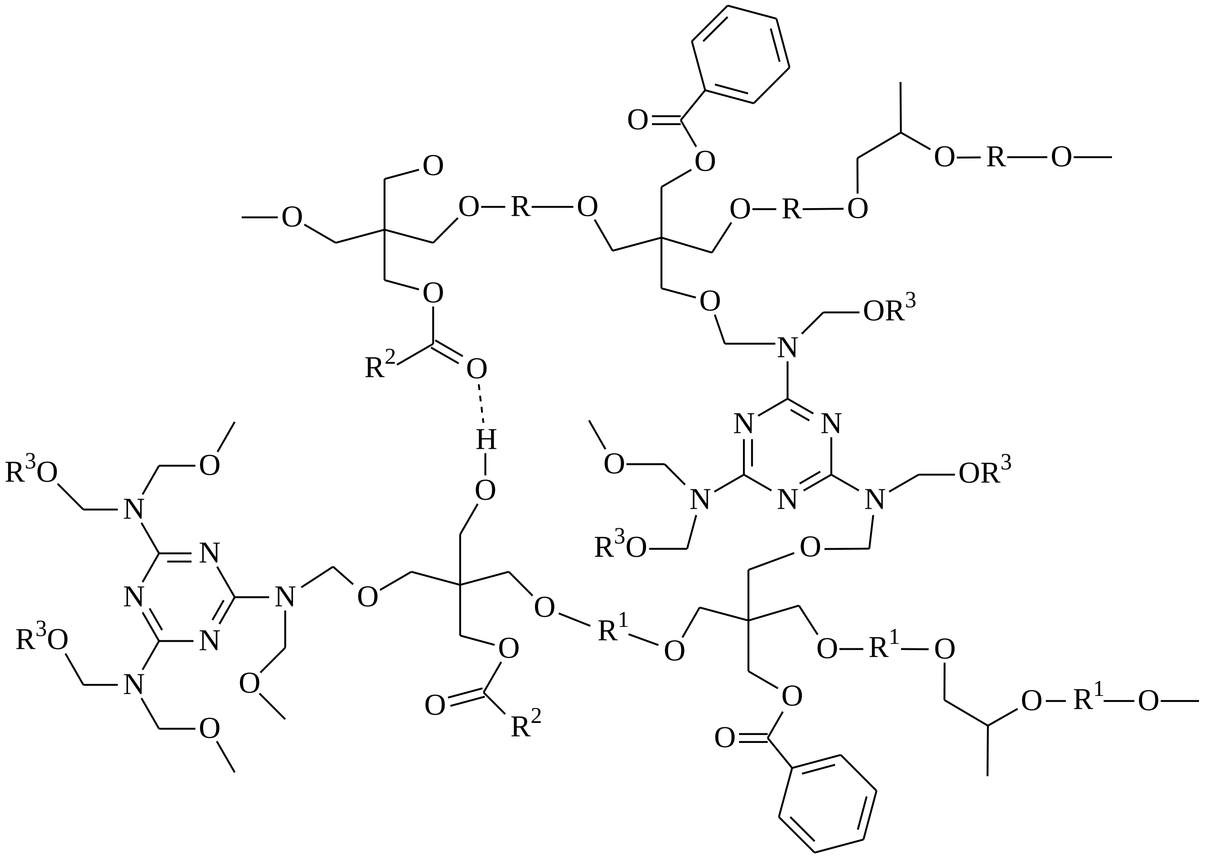


Figure 3. Possible cross-linking structure in a polymer obtained by mixing alkyd resin with melamine resin.

Besides the variation of the main components (resins) and their compositions, another method currently used to improve the properties of polymeric final materials is the addition of certain compounds. On the recent years, the addition of nanoparticles (NPs) has been commonly tested for the preparation of materials and the adjustment in material properties [13-15]. By mixing nanoparticles with polymeric matrix, the properties of modified polymer are substantially changed comparative with the polymer obtained by mixing with traditional additives [19].

The present study reports the response of several cured resins to thermal stress in electric rotary machines; the modifications of tested mixtures have been accomplished specifically by modifying the fatty component of alkyd resin. The optimal mixing ratio of the two components, alkyd and melamine resins, was evaluated. Additionally, several samples were modified by adding nanoparticles of Al_2O_3, SiO_2 and / or acrylamide in different proportions. Two oxides (e.g. Al_2O_3, SiO_2) and acrylamide were selected according to the literature data, which mentioned their useful properties in the increasing in insulating capacity of polymeric matrix [20]. The structural modifications of polymers and components obtained by this procedure were investigated by physicochemical methods, after their exposure to thermal and irradiation stress. Infrared spectra were recorded on solid state polymer samples with and without additives, subjected or not to thermal and/or irradiation stress. The chemiluminiscence technique was used to investigate the thermal behavior of cross-linked polymers.

2. MATERIALS AND METHODS

2.1. MATERIALS

The thermal stability study on the influence of mixture composition in cross-linked compounds was performed using three types of alkyd resins: alkyd resin based on sunflower oil, soybean oil and one resin containing equal proportions of sunflower oil and soybean oil. As curing agent, melamine resin in the form of iso-butylated poly(melamine-co-formaldehyde) solution was used. All three types of alkyd resins were purchased from a Romanian manufacturer, Rasin S.R.L., as uncured fluid mixtures in different solvents having the same content of phthalic anhydride (35 ± 1 %), pentaerythritol (8 ± 1 %), benzoic acid (1 ± 1 %), propylene glycol (16 ± 1 %) and vegetable oil (40 ± 1 %) in the nonvolatile fraction of the mixture.

Table 1. The components of mixtures used as alkyd resins in the curing process

Type of mixture	Solvents	Non volatile compounds concentration (w/w)
Mixture for alkyd resin 1: Sunflower oil, phthalic anhydride, pentaerythritol, benzoic acid, propylene glycol	toluene and butanol	55 ± 1 %
Mixture for alkyd resin 2: Soybean and sunflower oil, phthalic anhydride, pentaerythritol, benzoic acid, propylene glycol	xylene and butanol	60 ± 1 %
Mixture for alkyd resin 3: Soybean oil, phthalic anhydride, pentaerythritol, benzoic acid, propylene glycol	xylene and butanol	55 ± 1 %
Melamine resin Medium reactive iso-butylated melamine formaldehyde resin	isobutanol : xylene (7:1, w/w)	60 ± 1 %

Evonik Degussa provided silica (SiO_2) nanoparticles generally used as inorganic fillers for paints as condensed particles (trade name Aerosil 380) with a specific surface of 380 m^2/g and particles diameter in the range 3 - 15 nm was used as well. Alumina (Sigma Aldrich) nanoparticles with size in the range 30 – 60 nm and acrylamide (99 % purity, Merck) as curing agent were purchased.

2.2. METHODS

Insulating lacquer

The testing mixtures were prepared by mechanical blending of studied components. Alkyd and melamine resins were mixed and then, addition of fillers was performed. After blending, sample portions from each recipe were placed on a glass support as a thin layer with minimum thickness of 1 mm. Samples were cured by heating at 150 ^{o}C for 90 minutes. The time selected for maintaining to 150 ^{o}C is sufficient for volatile solvents to be removed and enough for the resins curing.

Thermal and radiation stress

The modifications appeared in cured resins by heat treatment simulating regular operation of rotary machines. Successive heating steps were applied, at 160^{o}C, 180^{o}C and 250 ^{o}C. The operation at these high temperatures may generate changes in chemical structure of polymeric lacquer that could be associated with reducing in the insulating capacity of metallic conductors. Mixtures consisting of alkyd resin, melamine resin and additives (acrylamide, added in proportions of 0.1, 1, 3, and 5 % (w/w), inorganic nanoparticles of SiO_2 0.5 % and Al_2O_3 0.5 % (w/w) were exposed to gamma radiation in an irradiator (Gammator M38 / USA), provided with a ^{137}Cs source. Irradiation was performed at room temperature at the dose of 50 kGy for all samples.

IR Spectroscopy

The study of structural modifications occurred in the polymeric samples was done by analysis of the infrared spectra [21-24] recorded on a Bruker Vertex 80 Fourier transforming IR spectrometer, equipped with an attenuated total reflection (ATR) device with reflexive diamond crystal. The scanning of each sample was performed in the wavenumbers range of 4000 cm^{-1} to 400 cm^{-1}.

IR spectra of coatings were registered for the different cured alkyd resins, melamine resin and the mixtures obtained by combining alkyd resin with melamine resin in the following ratios: 1:1, 3:1, 5:1, 7:1 and 10:1 (non-volatile alkyd resin: non-volatile melamine resin, w/w). Alkyd resins used in this experiment had the same composition of main components, but the type of oil is different from one formulation to the other. It is known that the type of oil used influences the dry time and performance of final cured resin [25].

Fatty acids could be classified into drying, semidrying and non-drying types. Non-conjugated oils added to alkyd resins are considered drying oils if their drying index, calculated as below, is higher than 70. The higher the amount of linolenic and linoleic content, higher drying index results [25].

Table 2. Fatty acids content in soybean and sunflower oil [26, 27]

	Saturated fatty acids	**Monounsaturated fatty acids**	**Polyunsaturated fatty acids**	**Linoleic acid**	**Linolenic acid**
	g / 100 g	**g / 100 g**	**g / 100 g**	**g / 100 g**	**g / 100 g**
Soybean oil	13.5 ± 0.93	28.5 ± 1.2	57.5 ± 6.5	49.5 ± 6.5	8 ± 3.4
Sunflower oil	8.8 ± 0.8	31.5 ± 4.5	60.0 ± 7.5	59.5 ± 7.5	-

Chemiluminescence technique

The stability evaluation of samples to thermal and radiative stress, was carried out with Lumipol 3 chemiluminometer. This instrument was firstly used in the isothermal mode, when the intensity of chemiluminescence (CL) radiation was recorded as a function of time, the temperature being constant. Samples of around 10 mg were weighted, within an analytical error of 10^{-4} g, on 1 cm diameter round aluminum trays. Then the curing thermal treatment (150^{o}C for 90 minutes) was applied.

After the curing stage a part of samples were subjected to thermal stress, and another part was subjected to γ-irradiation at 50 kGy. Isothermal spectra were recorded by measuring the intensity at 180^{o}C minute by minute. The accuracy of temperature measurements was ± 0.5 ^{o}C. The samples subjected to irradiation stress were immediately measured after the end of irradiation procedure, because the intensity drops quickly involving the decay of short life radicals.

For the comparison analysis, a blank sample was prepared with the following composition (w/w %): melamine resin 22 - 23 %, epoxy resin 1 % and alkyd resin up to 100 %. Epoxy resin was added to initially tested alkyd-melamine mixture for obtaining a better adherence to solid surface. After the blank preparation, the three fillers (acrylamide and nanoparticles of SiO_2 and Al_2O_3) were added. Modified samples were then cured by heating them for 90 minutes at 150^{o}C. After curing, samples were subjected to radiative and thermal stress. Finally, they were tested by chemiluminescence method.

3. RESULTS AND DISCUSSION

3.1. IR study of cured resins used as insulating coatings in rotatory machines

Even though the tested mixtures had different fatty acid compositions (table 2), the three types of alkyd resins used as a starting point of the study showed similar IR spectra after curing at 150^{o}C, as well as after further heating at 180^{o}C, indicating that a larger quantity of unsaturated fatty acids did not induce a higher tendency to oxidation.

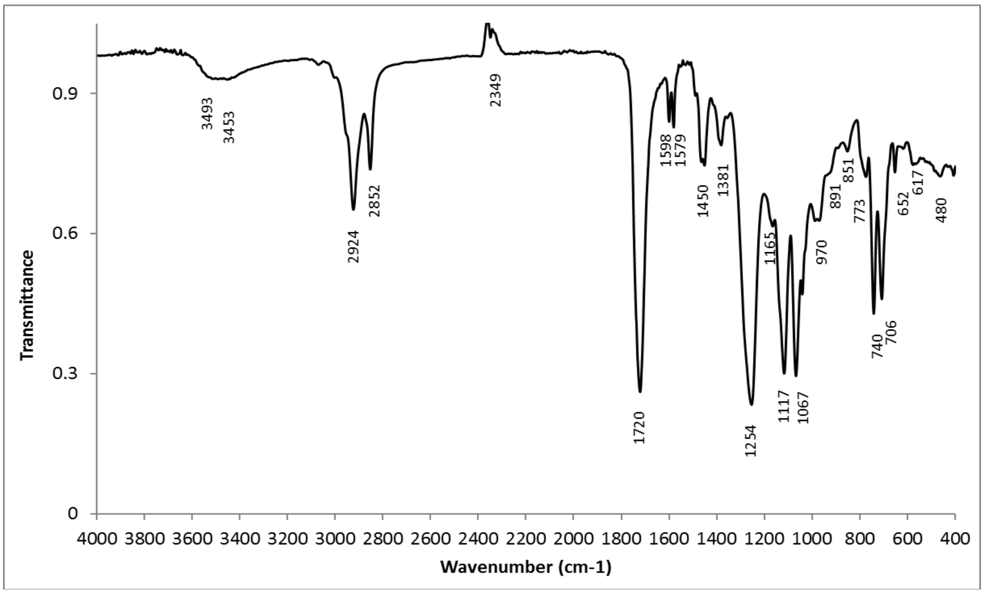


Figure 4. IR spectra of cured alkyd resins.

The heating of cured lacquer at 180 °C did not cause modifications in the recorded IR spectra. This behavior is an indication that the crosslinking process occur in the first heating step, at 150 °C. The maintaining of metallic conductor coatings in an oven at 150°C during one and a half hour insures the formation of a film with good insulating properties according to our results.

IR spectra of alkyd resins present a broad band of absorption with low absorption in the region 3600 – 3400 cm^{-1} ascribed to stretching frequencies of hydrogen bonded O-H. This fact, correlated with the signal at 1720 cm^{-1}, the stretching vibrations of the C=O, may suggest the presence of a small number of unreacted O-H remained in the polymeric matrix. The C-H stretching vibrations are differentiated in the spectrum by the characteristic absorption bands appeared at 2924 and 2852 cm^{-1}. Stretching vibrations at 1598 cm^{-1} suggest C=C bonds from linear alkenes, while absorption bands at 1579 cm^{-1}, 1450 cm^{-1} and 1381 cm^{-1} are present mainly as the characteristic of stretching vibrations of aromatic C=C bonds. It may also indicate the influence of bending vibrations of C-H with sp^3 hybridized carbon atoms.

The absorption band at 1254 cm^{-1} indicates the presence of C-O bond stretching corresponding to the esters of benzoic acid that exist in the structure of melamine resin, while absorption bands that appear at 1117 cm^{-1} and 1067 cm^{-1} point out the stretching vibrations of C-O bonds from free alcohol groups.

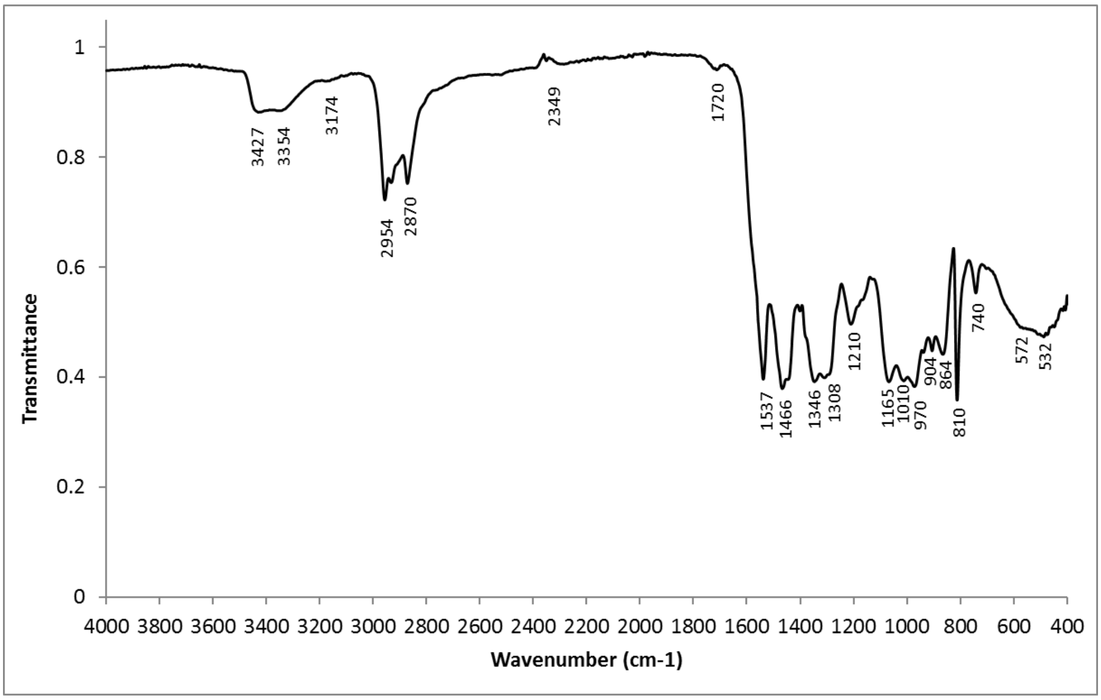


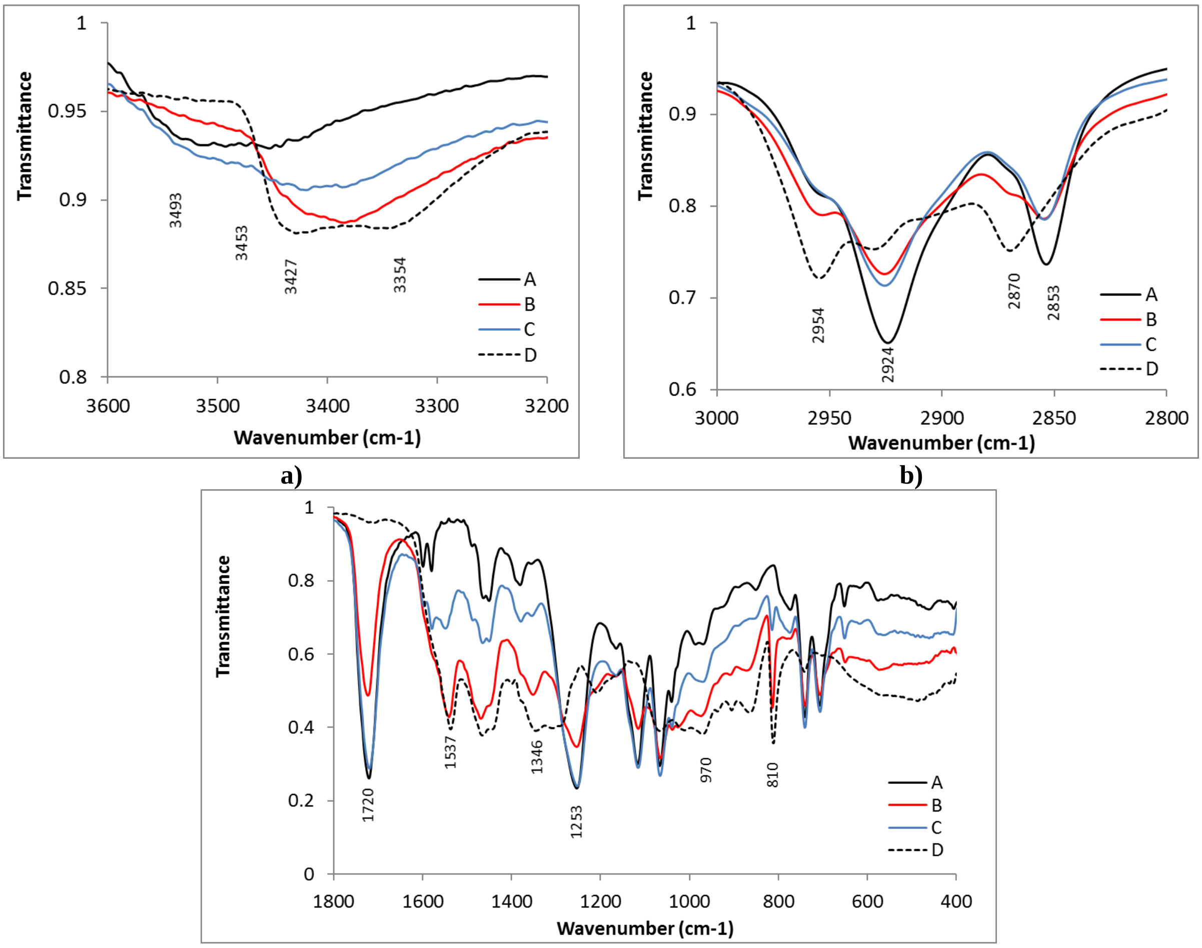
Figure 5. IR spectra of cured isobutylaled melamine resin.

IR spectra of melamine resin has a broad band near 3400-3300 cm^{-1}, with weak signal intensity, indicating the possible N-H stretching vibration in primary aryl amines and stretching vibration of O-H bond. The low intensity of the signal could be correlated with small number of N-H bonds relative to the initial N-H bonds from the reacting mixture. The reduced number of unreacted amino groups in the resin structure can be correlated with the wavenumbers of amino-substituted triazines, which would show specific absorption band in the region 1680-1640 cm^{-1}. However, this band does not appear in the IR spectrum recorded on cured melamine resin at 150°C. The similarly missed peak was observed for cured melamine resin at 180°C. The presence of the triazinic ring is indicated by the absorption band observed at 2954 cm^{-1} corresponding to the specific stretching vibration frequency of C-

H bonds with sp^2 hybridized carbon atom, as well as by the specific ring stretching vibrations observed at 1537 cm^{-1}, 1346 cm^{-1} and 970 cm^{-1}. The absorption band from 810 cm^{-1} corresponds to out of plan bending vibrations of C-H bonds. The weak signal recorded at 1720 cm^{-1} (specific band for C=O group) could be another indication of a small aldehyde quantity still not involved in the crosslinking process of melamine resin [7].

The behavior of prepared testing mixtures was compared with alkyd and melamine resins alone. Thus, alkyd and melamine resins individual samples were blank samples for further comparison, after they were subjected to the crosslinking process for 90 minutes heating at 150°C. Sample blanks and testing mixtures were thus treated according to the same procedure. A set of samples were heated for 120 minutes at 180°C in order to observe a further heating at a higher temperature is associated to further crosslinking process. Structure modifications that may be related to this process were evaluated.

Several preliminary tests were accomplished for defining the optimal mixing ratio range (w / w). The optimal mixing range relative to alkyd resin concentration covers the values from 50 to 100 % (w/w). If the alkyd resin proportion falls below 50 % (implicitly concentration of melamine resin rises over 50 %), the mixture exhibits the properties and behavior of the major component. It was observed that melamine resin is not adherent to solid surfaces and the heating leads to a brittle cured resin, while the polymer based on alkyd resin, obtained by the same curing procedure, showed a good flexibility.



a) b)

c)

Figure 6. Compared IR spectra of cured resins obtained by crosslinking alkyd resin, melamine resin, and their mixture: A – alkyd resin, B – mixture 1:1 (alkyd : melamine, w/w), C – mixture 10:1 (alkyd : melamine, w/w), D - melamine resin.

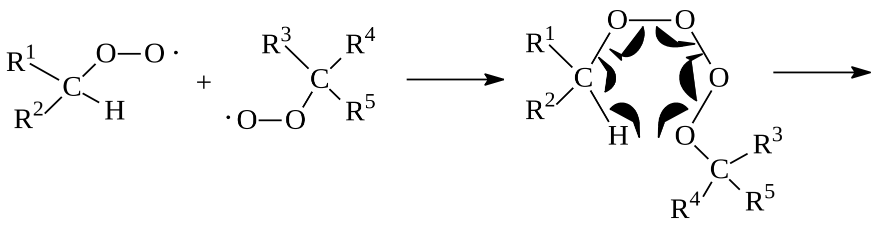
A next step of this study was the identification of an optimal ratio between the alkyd resin and the curing agent (melamine resin). The recorded IR spectra of the testing mixtures were compared with the blank spectra, the samples being prepared from alkyd and melamine resins as it was early mentioned. This comparison showed absorption peak shifts (Figure 6, a) - c)).

Figures 6 a - c show extended sections from the IR recorded spectra regions of absorption frequencies of 3600 to 3200 cm^{-1}, 3000 to 2800 cm^{-1} and 1800 to 400 cm^{-1}, respectively. The individual components (alkyd resin – line A, melamine resin – line D) and two of the studied mixtures, B – mixture 1:1 alkyd to melamine resin and C – mixture 10:1 alkyd to melamine resin were considered for comparison. In the Fig. 6 a), the specific shape of melamine shows two peaks at 3424 $cm^{-1}$and 3354 cm^{-1}. The mixtures B and C show similar signals at the same frequency range, but with a broader absorption band with a maximum at 3380 cm^{-1} that may include the stretching frequencies of O-H and secondary amines N-H. This can be correlated with the conversion of primary amines into secondary amines in the curing process. The absorption of the IR radiation at this wavenumber is higher for higher melamine resin content. In Fig. 6 c), the specific absorption frequencies for triazinic ring (1537 cm^{-1}, 1346 cm^{-1}, 970 cm^{-1} and 810 cm^{-1}), as well stretching vibration frequencies of C=O bond and C-O bond stretching frequency (1253 cm^{-1}) characteristic of benzoates can be noticed [24]. The examination of C=O stretching absorption at 1720 cm^{-1} and 1253 cm^{-1} suggests that the alkyd resin content influences the signal intensity. The higher the alkyd content in the mixture, the higher the absorption signal is recorded. A similar conclusion may be drawn in respect to the absorption band at 810 cm^{-1}; the higher the melamine content in the polymeric mixture, determines the higher the intensity of the absorption peak.

From the IR investigation, practical information could be drawn. By varying the melamine resin content in the polymeric mixtures, the obtained material tends to a structure similar to cured melamine resin when the concentration of this component is over 25% w/w (ratio 3:1 alkyd : melamine, w/w). If the melamine concentration falls below 20 %, the IR spectrum of the cured mixture tends to have the profile of the alkyd polymer. These data show that the optimal insulating material combines the properties of alkyd resin and of the melamine resin when the added melamine component reaches the proportion of 20 – 25 % (w/w).

3.2. Thermal and radiative stability evaluation of alkyd resins based coatings by chemiluminescence method

The measured chemiluminescence intensity is proportional with the concentration of the peroxide groups formed in oxygenated intermediates involved in the oxidation process [29]. The intermediates emit energy as electromagnetic radiation (photons), when they drop from an excited onto a lower energy level. Thus, the ability to measure the intensity of the chemiluminescence emission occurring while certain materials are heated is a useful tool in the evaluation of their stability for various applications and environments (Fig.7).



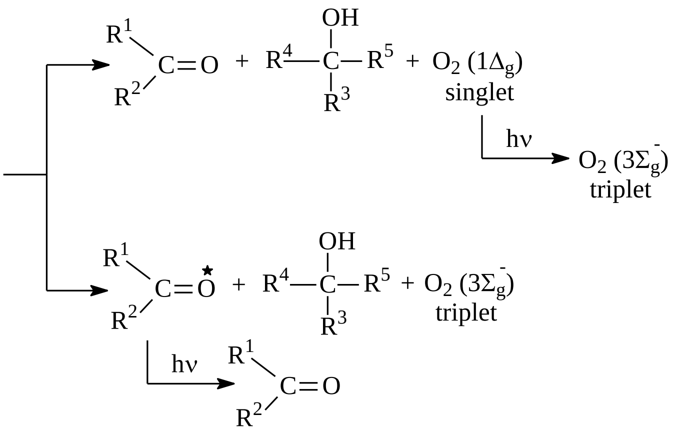


Figure 7. Scheme of process accompanied by emission of chemiluminescence radiation (Russel mechanism) [29].

One part of the samples (polymeric mixtures with and without additives) were first irradiated with a ^{137}Cs source under a dose of 50 kGy. After the radiative treatment, samples were tested in isothermal mode to determine the intensity of emitted chemiluminescence radiation. For the polymers free of additives, which were not subjected to thermal or radiative stress, the chemiluminiscence radiation intensity measurements show a fast decrease of the signal in the first minutes of heating at 180°C. After about 10 minutes, decreasing rate of the chemiluminescence/CL signal is lowering until it reaches an equilibrium, when the measured signal intensity reaches a plateau.

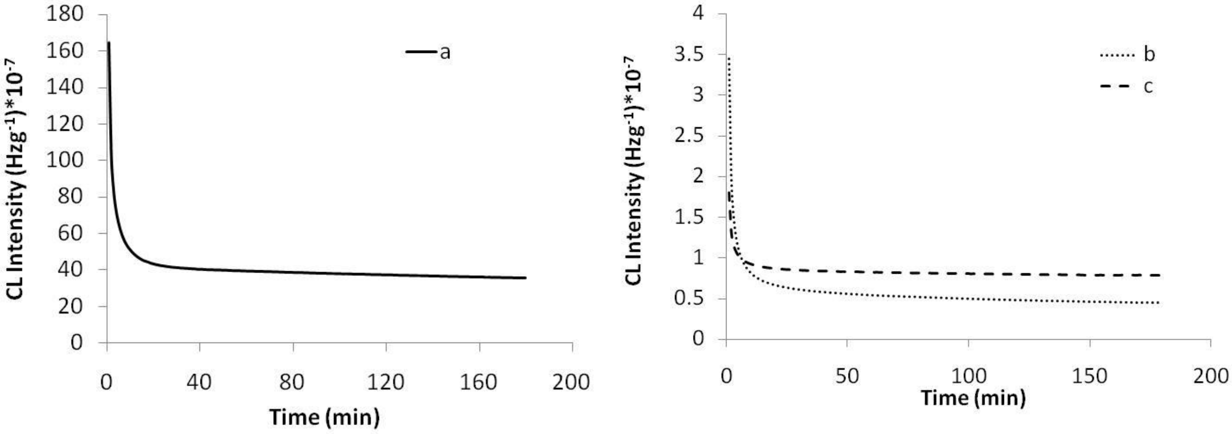
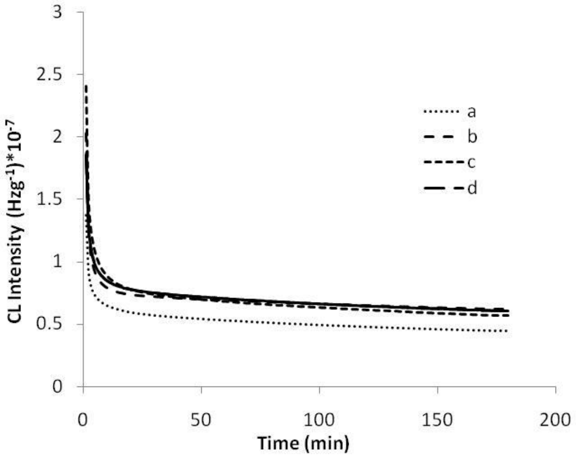


Figure 8. Chemiluminiscence intensity (isothermal mode – 180 °C) for lacquer without additives: a) lacquer, b) irradiated (50 kGy) lacquer, c) lacquer heated at 180 °C for 2 hours / 120 minutes.

Comparing the three curves in Fig. 8, it may be observed that recorded CL signal shows very high values for blank sample (cured polymer not subjected to heat or irradiation treatment). For the lacquer that was subjected to thermal or oxidative - radiative stress, small values of CL signal were recorded.

The cured resins with different fillers showed the same rapid decay in the signal intensities, the shape of the curve I_{CL} – time (chemiluminescence intensity vs. time) being the same regardless the additives used (Fig. 9). A small difference may be remarked. The resins with acrylamide presented low CL intensity (Fig. 9a) relative to the resins filled with Al_2O_3 or SiO_2 nanoparticles (Figs. 9 b - e). The addition of inorganic nanofillers led to high interface contact. This could bring a certain stability to resins, due to the absorption capacity of the interface to free radicals [30, 31].



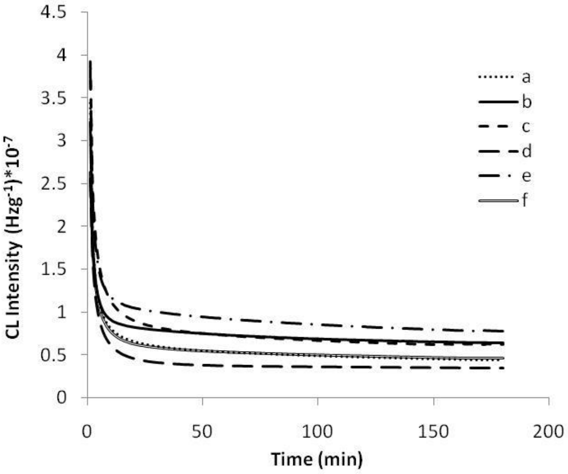


Figure 9. Chemiluminiscence intensity (isothermal mode – 180 ^oC) for lacquer with additives: a) acrylamide 3%, b) Al_2O_3 0.5 %, c) SiO_2 0.5 %, d) Al_2O_3 0.5 % + acrylamide 3 %, e) SiO_2 0.5 % + acrylamide 3 %

Figure 10. Chemiluminiscence intensity (isothermal mode – 180 ^oC) for irradiated lacquer (50 kGy) with additives: a) no fillers, b) acrylamide 3 %, c) SiO_2 0.5 %, d) Al_2O_3 0.5 %, e) Al_2O_3 0.5 % + acrylamide 3 %, f) SiO_2 0.5 % + acrylamide 3 %.

A different behavior of irradiated samples and thermal stressed samples was noticed. For the irradiated samples the CL signal in the first minute after stress action (irradiation with a dose of 50 kGy or heating to 180 ^oC) was higher and followed by a slower decrease than for thermally stressed samples. For the irradiated samples, the inflexion point of the CL intensity – time graphs are placed after 10 minutes of heating, while for the other samples (with and without thermal treatment) it can be found after 5 minutes. This experimental finding may be explained by existence of a high number of groups excited by irradiation. These groups tend to reach a lower energy level by emitting a quantum of electromagnetic radiation; thus, a longer time for the decay of the CL signal is recorded.

4. CONCLUSIONS

The effect of alkyd resin composition on thermal stability of lacquers used as coatings for electrical insulators was studied using IR spectroscopy and chemiluminescence technique. The main component of crosslinked polymers was alkyd resin and iso-butylated poly(melamine-co-formaldehyde) solution was added as curing agent.

IR Spectra of alkyd resins were similar in the three situations, where the fatty fraction was modified by adding soybean oil, sunflower oil or the 1:1 (w/w) mixture of the two oils indicating that a larger quantity of unsaturated fatty acids did not induced a higher tendency to oxidize.

The CL study showed that adding inorganic nanofillers to resins adds stability to thermal oxidative stress, due to the capacity of the large interface to absorb free radicals as confirmed by other studies.

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