

Applicability of Different Bio-based Polymers for Wiring Boards

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Abstract

The paper gives a review of experiments for the application of biodegradable, sustainable polymers as a wiring board material. In the paper two different biobased materials and the standard PCB materials FR4 and FR2 were compared. The investigations refer to mechanical, electrical performance, surface quality, flammability and applicability for Polymer Thick Film Technology (PTFT) of the biobased material. The biobased materials are polylactic acid (PLA) mixed with different contents of cellulose acetate (CA) or flame retardant zinc pyrophosphate (ZnPP) and polyurethane (PU) with CA. The results show that the addition of CA to the polymers leads to a change of different properties for PLA and to a significant change in mechanical properties for PU. The use of ZnPP in PLA shows major improvements regarding the flammability of the polymer. The properties of the samples are in the same order of magnitude as the properties of FR2.

Keywords

Eco-Electronics, polyurethane, polylactic acid, wiring board

1 Introduction

The electronic waste problem is becoming more and more important. The amount of produced electronics, e-waste and also the increasingly short life of electronics contribute to the waste problem. Therefore a possibility to recycle should have been detected. The recyclability is the ability of a material to be captured and separated from a waste stream for conversion or reuse. The standard material is not recyclable due to the flame retardant polybrominated diphenyl ethers. Because of the recyclability or the degradability, the investigations of bio-based wiring board materials are important and could be an alternative for FR4. Bio-based materials are materials derived from biological or renewable resources and offer the possibility for recycling. Polylactic acid (PLA) could be a good alternative, because of the good characteristics, great chemical versatility and is counted among the renewable resources. It offers also the possibility of biodegradation. Biodegradation is the ability to break materials into basic components e.g. carbon dioxide or methane [1]. Our previous work focused on PLA with different contents of cellulose acetate (CA) and aluminum polyphosphate and found out that it is critical to use conductive pastes because of the thermal stress [2, 3]. Géczy et al.

[4] investigated the mechanical performance of pure PLA and cellulose acetate. They found out that both materials act weaker than standard materials and that additives are needed to improve the mechanical performance. Mattana et al. [5] investigated the use of PLA in organic electronic devices and for sensor applications [6]. It was also investigated that PLA can be used as a substrate material for antennas for 2.45 GHz ISM [7]. Oi et al. [8] increase the crystallinity of PLA from 6 % to 42 % by performing a heat-treatment of PLA before the application to reduce the problem of thermal stress for PLA. The effects of a decreasing crystallinity of PLA due to increased temperature have been investigated as well [9, 10]. Different additives were tested to improve the properties of PLA [11]. Jute fibers were used to improve the mechanical properties. Kenaf, cellulose and wood fibers were tested as well [12, 13]. As a possible halogen free alternative different phosphorus based flame retardants like aluminum polyphosphate have been tested [11, 14].

In the present paper another partially renewable material was tested. Polyurethane (PU) could also an alternative for wiring board material. In this case renewable

means the relation to a natural resource and the use of raw materials that are produced from regrowing substances. The main concern of those biopolymers is to improve their characteristic profile to reach that of standard materials. Like Géczy et al. [4] stated PLA has to be modified with additives. Therefore this paper deals with PLA mixed with different contents of cellulose acetate and zinc pyrophosphate (ZnPP). Zinc pyrophosphate (ZnPP) is a halogen free alternative flame retardant [15].

The paper shows an overview of the mechanical and electrical performances of PLA/CA and PU/CA compounds in reference to wiring board materials. The present paper deals with production, processing and application of these bio-based materials.

2 Method and materials

2.1 Investigation strategies

Previous studies showed that PLA is a suitable material for wiring boards in limited applications. PLA can be obtained from renewable resources. Certain additives, like fillers or flame retardants, have different effects on the properties of PLA. In this study CA fibers were used as a filler material to improve the mechanical properties of PLA. To improve PLA's flame behavior ZnPP as a flame retardant was added. Because of the relatively low glass transition temperature of PLA its applicability is still limited. A different biopolymer partially based on renewable resources was produced and characterized [16]. The comparison of different properties of PLA composites and PU composites is shown in the following paper.

2.2 Material and variations

The PLA used was supplied by NatureWorks (USA) under the trade name Ingeo™ Biopolymer 3251D. The CA (CA-398-30) used was supplied by Eastman Chemical Company (USA). The zinc pyrophosphate is found as Budit 3178 and was supplied by Budenheim Ibérica S.L.U. (Spain). For the PU synthesis the bio based polyether polyol Lupranol/2005/1/Balance from Basf SE (Germany) was used. TIB KAT 218 from TIB Chemicals (Germany) was used as a catalyst. 1,4 Butandiol was supplied by KMF Laborchimie Handels GmbH (Germany) and dried over molecular sieves 3 Å before use. Basonat H (hexamethylene diisocyanat) was supplied by Basf SE (Germany).

The sample preparation of the PLA composites took place via melt compounding. For this PLA pellets were inserted in a stainless steel bowl on a hot plate with a thermostat. The PLA pellets were continuously stirred

and melted. The required amounts of CA or ZnPP were added to the melted PLA while stirring continued. After reaching a sufficient homogeneity of the melted composite material, it was transferred on a PTFE mould to cool down. To shape the composites the material was melted in a silicone mould in an oven with recirculating air.

The preparation of the PU samples took place via a one shot process. The Polyol, the chain extender Butandiol and the catalyst dibutyl tin dilaurate were carefully mixed in a stainless steel cup. The required amount of CA was carefully stirred in the mixture. The mixture was tempered at 85 °C and 500 mbar for 1 h in a vacuum drying oven. The required amounts of isocyanate were carefully added to the mixture. The mixture was stirred and transferred into a silicon mould. It was cured at 85 °C for 24 h. The surface of the samples was grinded down to receive a level surface with the polishing machine from Struers RotoPol-31 and RotoForce-4 with a grain size of 120, a force of 100 N and 300 rpm. Table 1-3 show the compositions of the samples.

2.3 Surface energy

A different method to gain knowledge of the adhesion on a printable surface is the wettability. By measuring the contact angle of a liquid on the surface of the sample

Table 1 Composition of the PLA/CA samples

Sample	PLA [g]	CA [g]
PLA/CA1	100	1
PLA/CA2,5	100	2.5
PLA/CA5	100	5
PLA/CA10	100	10
PLA/CA15	100	15
PLA/CA20	100	20
PLA/CA25	100	25
PLA/CA30	100	30
PLA/CA35	100	35
PLA/CA40	100	40

Table 2 Composition of the PLA/ZnPP samples

Sample	PLA [g]	ZnPP [g]
PLA/ZnPP1	100	1
PLA/ZnPP5	100	5
PLA/ZnPP10	100	10
PLA/ZnPP15	100	15
PLA/ZnPP20	100	20
PLA/ZnPP25	100	25
PLA/ZnPP30	100	30
PLA/ZnPP35	100	35

Table 3 Composition of the PU samples with the weight of the different components

Sample	Component weight [g]			
	Polyol	Butandiol	CA	Basonat H
PU	35	2.19	0	7.40
PU/CA5	35	2.19	1.86	7.75
PU/CA10	35	2.19	3.72	8.10
PU/CA15	35	2.19	5.58	8.46
PU/CA20	35	2.19	7.44	8.81
PU/CA25	35	2.19	9.30	9.71
PU/CA30	35	2.19	11.16	9.52
PU/CA35	35	2.19	13.02	9.88
PU/CA40	35	2.19	14.88	10.23

material, the surface energy of the material can be calculated. According to the Owens, Wendt, Tabel and Kaelble (OWRK) theory the surface energy consists of disperse and polar interactions. The polar interactions are caused by dipole-dipole interactions and hydrogen bonds. The dispersive interactions are caused by van der Waals interactions. For the measurement of the contact angle the OCA Contact Angle System from DataPhysics was used. The test liquids were deionized water, diiodmethane and ethylene glycol. The surface energy of the test sample can be calculated from the contact angle and the surface tension of the test liquids. The contact angle of every liquid was measured ten times and a mean value was calculated.

2.4 Three-point bending

One import mechanical requirement for a wiring board is the stiffness of the material. In order to increase the reliability of electronic components and conducting paths, the materials should be used for different applications depending on their level of stiffness. Materials with a high stiffness should be used for stiffness applications and materials with a low stiffness for flexible applications. Therefore the three-point bending test was applied to determine the bending strength and to determine the possible application field of the substrate. Three-point bending tests according to DIN EN ISO 14125 standards for fibre-reinforced plastics were performed.

With this measuring method the stress-strain behavior could be determined and subsequently the E-modulus calculated. The measurement setup consist of two supports with an radius of R_2 , and a distance L (span), which are arranged parallel. The compression is placed in the center between the supports and has a radius of R_1 . The test specimen is located between the supports and the compression.

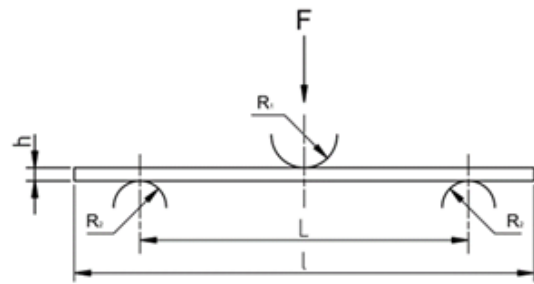


Fig. 1 Schematic of three-point bending test setup

The compression applies a certain force onto the substrate. For the samples a thickness h of 2 mm was defined. According to DIN EN ISO 14125 the samples should have a width b of 25 mm, the length l of 40 mm and the span L should be 32 mm. The supports had a radius of 5 mm. The bending speed was 0.0143 mm/s. To calculate the E-modulus the following formula has been applied

$$E_f = L^3 / (4bh^3) (\Delta F / \Delta s) \tag{1}$$

F is the applied force at s' and s'' , s is the deflection at strain ϵ_f and ϵ''_f :

$$s' = (\epsilon_f L^2) / 6h \tag{2}$$

$$s'' = (\epsilon''_f L^2) / 6h \tag{3}$$

2.5 Dielectric Strength

The electrical breakdown is the loss of insulating properties of the polymer because of electrical influence. In accordance to the dielectric strength the insulating ability of polymers can be concluded. The measurements were carried out according to IEC 60243. The measurement setup consists of a container filled with vegetable oil. In the middle of the container are two ball electrodes with a diameter of 20 mm. Vertical and at the same axis between the electrodes the specimen with a geometry of 75 x 75 x 2 mm was mounted (Fig. 2). The voltage was continuously increased with a constant slew rate until breakthrough beginning at 0 V.

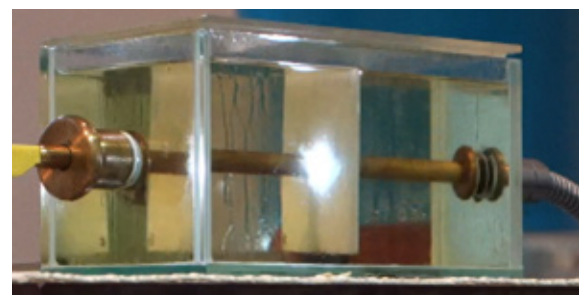


Fig. 2 Measurement setup to measuring the Dielectric Strength

Polymers have good electrical insulating properties. These electrical insulating properties of materials can be described with the permittivity. The measurements have been performed according to IEC 60250 with a schering bridge method. The specimen is placed between the plates of a capacitor and DC voltage is applied. The reference is an air filled capacitor. The used specimen geometry is 150 x 150 x 2 mm.

2.6 Surface resistivity and Volume resistivity

Additional electrical characteristics are surface resistivity and volume resistivity of polymer materials. These characteristic values are aimed at evaluating the insulation capability. The surface resistivity is the quotient of applied voltage between two electrodes and the current between these electrodes. Both electrodes are positioned on the same surface. The DC voltage is applied within a certain period of time. On the surface of the test specimen, which is level and coplanar, two circular, concentric plate electrodes with a diameter d_1 , distance between the ring electrode and the inner electrode g and p the circumference of electrode were positioned. Specific voltage and constant current determine the resistance value R_{OX} . For improved comparability, the specific surface resistivity σ_o is used [17].

$$p = \pi(d_1 + g) \quad (4)$$

$$\sigma_o = p/g \cdot R_{OX} \quad (5)$$

The volume resistivity ρ_D is the quotient of the applied voltage between two electrodes and the current between these electrodes. The electrodes with an area A_E and R_{DX} is the measured resistance are positioned on opposite sides of the polymer material [17].

$$A_E = \pi/4 \cdot d_1^2 \quad (6)$$

$$\rho_D = A_E/h \cdot R_{DX} \quad (7)$$

where h is the thickness of specimen.

2.7 Compatibility of PTFT

For the application as a wiring board lead structures are needed. To realize this the polymer thick film technology was used. The screen-printer DEK 249 and the conductive paste DuPont 5015 were used. The curing took place in a heat cabinet. To measure the conductivity of an electrical conductive layer, the sheet resistance of a silver conductive paste on PLA/CA, FR4 and PU were compared. The sheet resistance is calculate in accordance to the following formula.

$$R_F = R_{FX} \cdot b_L/l_L \quad (8)$$

where R_F is the sheer resistance, R_{FX} is the measured resistance with the relation of the width of the wire b_L and the length l_L . Because of the relatively low glass transition temperature of PLA a curing temperature of 80 °C was chosen for all substrate variants. This investigation is done to figure out whether a curing temperature of 80 °C is sufficient for curing of silver conductive pastes.

3 Investigations and results

3.1 Investigations of surface properties and mechanical properties

3.1.1 Roughness

The standard material FR4 has a $Sa=2.62 \mu m$. All samples show higher roughnesses than FR4. The roughness of the PU compounds increases with higher CA content. The addition of CA to PLA leads to an increased roughness of the samples. It is interesting that this increase is already recognizable with low amounts of CA. A further increase of CA in the samples leads to a gradual decrease of the roughness. The decrease with higher CA content can be explained with the inhomogeneity of the samples. More CA fibres can be on the surface of the sample. A higher roughness suggests that the adhesive strength is better. This should be confirmed in further investigations. For PLA, less CA content is better for the processing to a wiring board.

3.1.2 Surface energy

The surface energy, the polar and dispersive interactions of the PLA and PU composites are shown in Table 1 and 2 respectively. It can be seen that the amount of added CA has no significant influence on the distribution of the surface

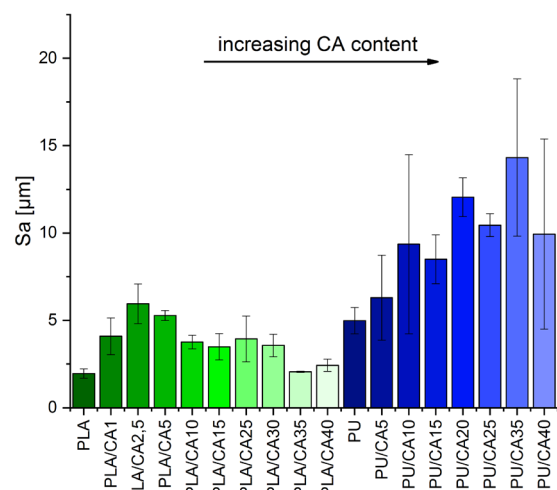


Fig. 3 Roughness (Sa) of PLA/CA and PU/CA composites

Table 4 Surface energy, dispersive and polar interaction of PLA/CA composites

Sample	Surface energy [mN/m]	Dispersive interactions [mN/m]	Polar interactions [mN/m]
PLA	42.18 ± 2.62	34.24 ± 2.19	7.94 ± 1.44
PLA/CA10	43.13 ± 1.91	38.19 ± 1.37	4.94 ± 1.33
PLA/CA20	44.47 ± 3.36	38.65 ± 2.75	5.82 ± 1.93
PLA/CA30	46.68 ± 2.13	44.54 ± 1.98	2.14 ± 0.79
PLA/CA40	44.66 ± 2.31	40.37 ± 1.95	4.29 ± 1.24

energy into polar and dispersive interactions. Rather the value of the surface energy and its distribution is influenced by the choice of matrix polymer. In both the PLA and PU composites the dispersive interactions outweigh the polar interactions. Which means that in both cases the van der Waals interactions have a greater influence on the surface energy than the different polar interactions. For a later lamination process of the polymer with copper foil it is important to know the surface energy and its distribution of the copper foil. Values of 34 mN/m for the surface energy, 28 mN/m for the dispersive interactions and 6 mN/m for the polar interactions of copper were found [17]. Because the magnitude and the distribution of the surface energies of the composites and copper are similar, the adhesion between copper and the polymers should benefit.

3.1.3 E-Modulus

Fig. 4 and 5 show the E-Modulus for the PLA composites and the PU composites respectively. The E-modulus of PLA decreased with increasing CA amount. Under the same condition, PU samples show a rising E-modulus with increasing CA amount. Both materials show an

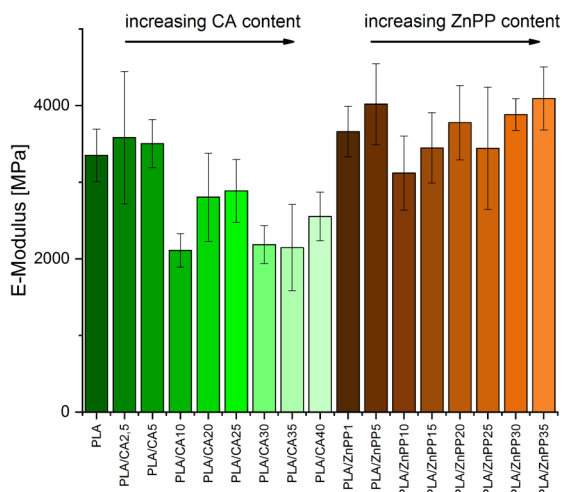


Fig. 4 E-Modulus of PLA/CA and PLA/ZnPP

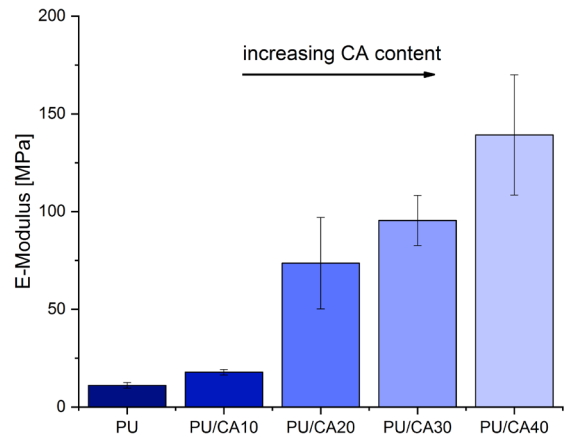


Fig. 5 E-Modulus of PU/CA composites

E-Modulus in different magnitudes. PLA demonstrates a good E-modulus for stiffness applications. From the sample PLA/CA10 forward variation of the E-modulus could be due to inhomogenities in the samples. During the production process it was observed that the viscosity of the compound increases significantly with the CA amount. This negative processability makes it more difficult to produce a homogenous compound. On the other hand, the E-Modulus of PU and its composites is significantly lower compared to PLA composites despite the addition of CA. The samples from PU/CA10 forward could be used as a flexible substrate. Here a similar aspect of inhomogeneity should be taken into account by interpretation of this data.

3.2 Electrical properties

3.2.1 Dielectric strength

Fig. 6 shows the dielectric strengths of the PLA composites. With different CA contents the dielectric strengths show nearly a constant behavior around 20 kV/mm. The

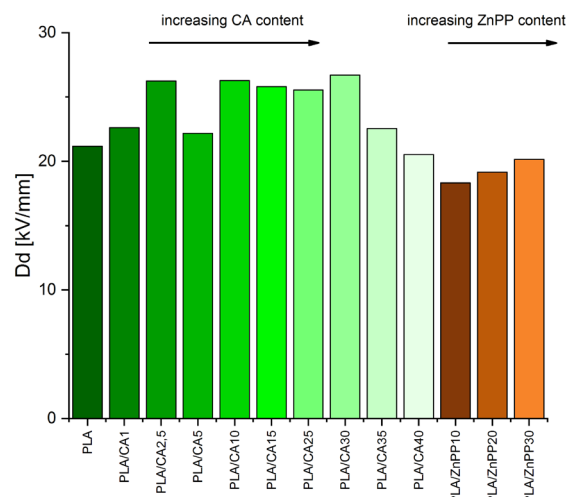


Fig. 6 Dielectric strength of PLA/CA and PLA/ZnPP composites

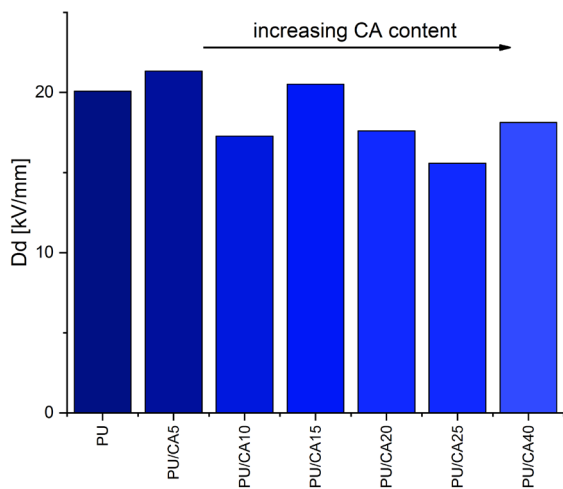


Fig. 7 Dielectric strength of PU/CA composites

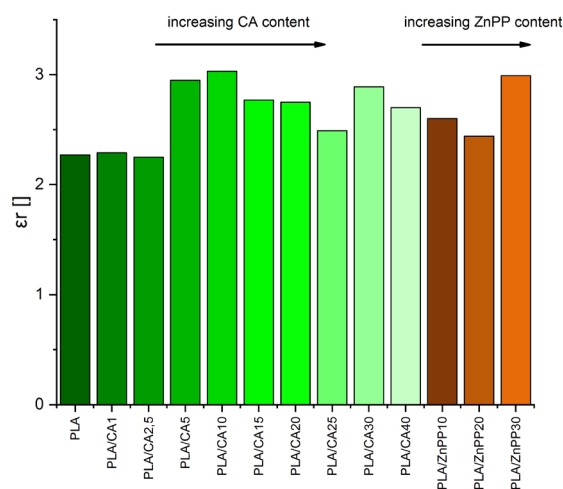


Fig. 8 Dielectric constant of PLA/CA and PLA/ZnPP composites

deviations could be caused by the inhomogeneities within the material. Because the samples were produced in lab scale inhomogeneities were unavoidable. In further investigations the effect should be FR4 shows a dielectric strength of 29 kV/mm and FR2 19.7 kV/mm. The values of PLA/CA are of similar magnitude compared to FR2 or even higher. The addition of ZnPP to PLA leads to a decrease of the dielectric strength.

For PU a similar trend is apparent. The variations may be caused by inhomogeneities and thus the unequal distribution of CA within the sample as well.

3.2.2 Dielectric constant

Fig. 8 shows the dielectric constant results of PLA CA and ZnPP composites. The first two PLA/CA samples with a very low CA content show no significant difference to pure PLA. From PLA/CA5 onwards the dielectric constant of the samples increases to a nearly constant value for different CA contents. But this can also be measurement errors. A similar effect is visible for PLA with ZnPP. The low permittivity indicates a negative loading of the material. The cause of the capacity in the material is the orientation polarization. For these substrate variations good results could be achieved. These results should be handled carefully because multiple measurements were not performed. FR4 shows a permittivity of 4.6.

3.2.3 Surface resistivity and Volume resistivity

Table 5 shows the measured surface and volume resistivity of selected PLA and PU composites as well as FR4.

For Interpretation, the magnitude is significant. Polymer materials need to have a resistivity of $> 10^{12} \Omega$

or rather $> 10^{12} \Omega/\text{cm}$ to be insulating. The results of PLA composites are in the range between 10^{14} and 10^{16} . PU composites are around 10^{15} . From that point of view both bio-based materials are suitable for wiring board material.

3.3 Compatibility on PTFT

Fig. 9 shows sheet resistance of silver paste on different substrates depending on the curing time. PU shows a more rapid decrease in resistance compared to PLA and FR4. This means that the curing temperature of 80 °C can be enough to cure a silver paste on PU substrates. The PLA composite shows better resistance values than FR4 as well. This suggests that at higher curing temperatures the conductive paste on PLA shows a similar good conductivity than on FR4. Nevertheless, the curing temperature of 80 °C is not high enough for sufficient conductivity.

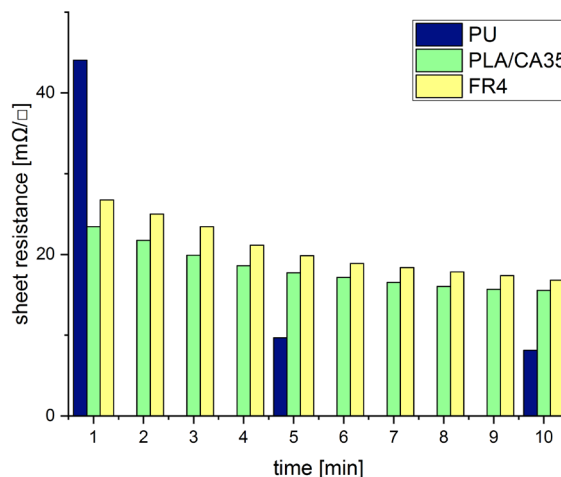


Fig. 9 Sheet resistance of the PLA/CA35, FR4 and PU

Table 5 Surface and Volume resistivity of PLA composites and PU composites

Sample	Surface resistivity [Ω]	Volume resistivity [Ω/cm]
PLA	$4 \cdot 10^{15}$	$1 \cdot 10^{15}$
PLA/CA1	$7 \cdot 10^{15}$	$5 \cdot 10^{16}$
PLA/CA2,5	$3 \cdot 10^{15}$	$1 \cdot 10^{17}$
PLA/CA5	$3 \cdot 10^{16}$	$1 \cdot 10^{17}$
PLA/CA15	$5 \cdot 10^{15}$	$5 \cdot 10^{16}$
PLA/CA25	$1 \cdot 10^{15}$	$3 \cdot 10^{16}$
PLA/CA30	$9 \cdot 10^{14}$	$1 \cdot 10^{17}$
PLA/CA35	$4 \cdot 10^{15}$	$2 \cdot 10^{17}$
PLA/CA40	$5 \cdot 10^{14}$	$1 \cdot 10^{17}$
PU	$4 \cdot 10^{14}$	$1 \cdot 10^{16}$
PU/CA35	$2 \cdot 10^{14}$	$2 \cdot 10^{15}$
FR4	$1 \cdot 10^{12}$	$5 \cdot 10^{12}$

Fig. 10 shows test prints on different substrate materials (PU, PU/CA35, PLA and PLA/CA35). All substrates show printable structures. It can also be seen that with increasing CA content the structures were more inexact, because of the increasing roughness of the materials. With a higher CA content, more CA fibers are on the surface. Therefore, it can be concluded that lower CA contents are better suited for screen-printing.

3.4 Flame protection

The fire behavior of the samples was tested with the help of a self constructed chimney that was build according to DIN 53438. The samples were fixed between two metal

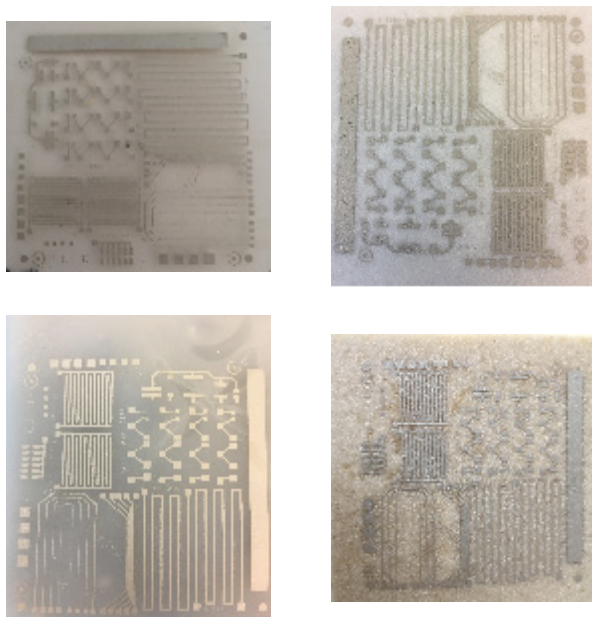


Fig. 10 Screen printing on PU, PU/CA35, PLA and PLA/CA35

plates inside the chimney. Through a hole in the wall a burner was positioned so the flame reaches only the bottom of the sample. After an inflammation of 3 s and after 7 s the observations of the samples were noted. Following this treatment an additional flame treatment was performed.

Table 6 shows the observations of the flame retardancy test. It was observed that the PLA samples start burning with melted substrate dropping down immediately after coming in contact with the burner flame. The conclusion that PLA is a fast burning material was confirmed [5]. CA fibers are easily flammable as well. Therefore, the addition of a flame retardant into the polymer is necessary to avoid easy burning. It was observed that the samples with ZnPP did not start burning during this test. Which meant a significant improvement compared to the pure PLA and the PLA/CA composite. Therefore the flame retardant effect that is necessary in PCB materials could be achieved with the addition of ZnPP into PLA.

4 Conclusion

This paper deals with the influence of CA and ZnPP as additives on the properties of PLA and PU. The roughness investigation lead to the conclusion that the roughness can already be increased with a small amount of CA. Especially the PU/CA samples showed a significant influence of the amount of CA on the E-modulus. Even though the E-modulus of PU composites increases with the amount of CA, the E-modulus of the composites was still low enough for them to be used for flexible applications. The E-modulus of PLA composites suggests the use in rigid applications. The results of different electrical properties show that these additives have no significant influence on them. The magnitudes of the electrical properties are similar to the properties of the FR2 material. The applicability of PTFT on the samples was tested and showed that the use is appropriable for both the PU and PLA substrates. PU samples with lower amounts of CA show better structure precision than with higher amount. Though the curing temperature of 80 °C needs to be increased in case of PLA. Testing the flame retardancy lead to the conclusion that the addition of a flame retardant like ZnPP is necessary. Overall both PLA and PU composites are suitable

Table 6 Results of flame retardancy test observation.

PLA/CA-composites	Material is burning with a lot of material dripping from the substrate during the fire test
PLA/ZnPP	Material is not burning, only smoulders with less material loss

for screen printing applications. The used additives lead to an improvement of the samples properties. PU composites of lower CA content should be preferred because the fibrous character of the CA negatively influences the quality of screen printing. Further investigations should include a method to increase the temperature stability. Additional

parameters that are relevant for the application of the materials as wiring boards should be tested.

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