Procesing '22

ZBORNIK RADOVA

35. Međunarodni kongres o procesnoj industriji

Holiday Inn, Beograd

1–3. jun 2022.

SMEITS

УНИВЕРЗИТЕТ У БЕОГРАДУ МАШИНСКИ ФАКУЛТЕТ

SET SAMIT ENERGETIKE TREBINJE

ZBORNIK RADOVA

pisanih za 35. Međunarodni kongres o procesnoj industriji
PROCESING '22

SMEITS

2022

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PROCESING '22

Holiday Inn, Beograd

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inženjera i tehničara Srbije (SMEITS)
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Procesing '22 organizuje Društvo za procesnu tehniku pri SMEITS-u, a u Naučnom i Organizacionom odboru prisutni su predstavnici svih Mašinskih fakulteta u Srbiji kao i Tehnoloških i drugih fakulteta u okviru kojih je oblast procesne tehnike zastupljena u nastavi.

Pomoć u organizovanju Procesinga '22 dali su članovi Katedre za procesnu tehniku Mašinskog fakulteta Univerziteta u Beogradu i mnogih drugih fakulteta iz Srbije.

Ovogodišnji skup završava se posetom novom Centru za upravljanje otpadom u Vinči.

U Beogradu
juni 2022.

SADRŽAJ

Procesne tehnologije

Projektovanje, izgradnja, eksploatacija i održavanje procesnih postrojenja

Inženjerstvo životne sredine i održivi razvoj u procesnoj industriji

Energetska efikasnost u procesnoj industriji

Procesi i postrojenja u pripremi i prečišćavanju vode u procesnoj industriji

Modelovanje i optimizacija procesnih i termoenergetskih postrojenja

Merenja i upravljanje u procesnoj industriji

Menadžment kvaliteta i standardizacija u organizacijama

Oglasni deo

KOMPOZITNI MATERIJALI NA BAZI NEZASIĆENIH POLIESTARSKIH SMOLA DOBIJENIH IZ BIOOBNOVLJIVIH IZVORA I OTPADNE KAFE

BIOBASED COMPOSITE MATERIALS OBTAINED FROM UNSATURATED POLYESTER RESINS AND WASTE COFFEE

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Upotreba bioobnovljivih monomera umesto stirena u proizvodnji nezasićenih poliestarskih (NZPE) smola, koje se u potpunosti dobijaju iz bioobnovljivih izvora predstavlja veliki izazov. Savremena istraživanja su ukazala da je stiren vrlo toksičan po ljudsko zdravlje i kategorizovan je kao potencijalno kancerogena supstanca. Upotrebom dimetil itakonata kao alternativnog rastvarača, koji se može dobiti iz obnovljivih izvora, obezbeđuje se niska viskoznost koja je neophodna za industrijsku primenu NZPE smola. U ovom radu je sintetisana NZPE smola na bazi ćilibarne kiseline, itakonske kiseline i propilen glikola, koja je zatim razblažena dimetil itakonatom. S obzirom da NZPE smole imaju značajnu primenu u izradi kompozitnih materijala, u ovom radu su dalje ispitivana svojstva materijala pri različitim udelima otpadne kafe kao punioca (10, 20, 30 mas.%). Nakon sinteze određena je viskozimetrija, mehaničke karakteristike kompozitinih materijala, kao i sadržaj gel faze. Strukturna svojstva ispitana su primenom FTIR spektrokopije .

Ključne reči: *materijali dobijeni iz bioobnovljivih izvora; otpadna kafa; itakonska kiselina; održivi razvoj*

Replacing styrene with biobased monomers to produce fully biobased unsaturated polyester resins (UPRs) turned out to be quite a challenging task. Styrene has been recognized as a hazardous and a potentially carcinogenic substance for human health. Using dimethyl itaconate as an alternative reactive diluent to styrene, which can be obtained from renewable resources, enables low viscosity for easy manipulation of mentioned UPRs. Fully biobased UPRs in question were based on itaconic acid, succinic acid, and propylene glycol, which were then diluted with dimethyl itaconate. Having in mind that UPRs can be used as a matrix for composite materials, this paper examines the effect of different weight ratios of spent coffee grounds as a filler. To determine the application of thus obtained composite materials, characterization consisted of examining mechanical properties based on viscosity measurements, uniaxial stretching experiments, FTIR spectroscopy and gel content analysis.

Key words: *biobased materials; waste coffee; itaconic acid; sustainable development*

1 Introduction

At the level of policy generation, the principles embedded in the circular economy are now emphasized in process and product design. This has introduced several visionary concepts i.e., the bioeconomy, the biobased society and the green economy that are now redirecting the strategic planning of majority industrial sectors.

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Application of waste materials in polymer industry is quite challenging, however it can lead to lower energy consumption, reduced waste generation and it can change the overall effect that industrialization has on the environment. Therefore, the search for sustainable, cleaner technologies that utilize waste materials is very important.

Coffee is present in every culture around the world. The abundance in consumption with the constantly growing population results in large amounts of waste generated. Coffee beans' annual production in the 2019/2020 crop year exceeded 10 million metric tons [1]. Since coffee production doesn't have significant amounts of by-products, it is implied that waste from beverage consumption is almost equal to the initial mass obtained from coffee beans [2].

There are a lot of environmental issues arising from the inadequate disposal of this waste material [3]. Spent coffee grounds (SCG) is the final product after brewing. SCG contain large amounts of organic compounds (i.e. fatty acids, lignin, cellulose, hemicellulose, and other polysaccharides) that justify its valorization [4]. Because of many valuable compounds found in SCG, researchers have found various application options for this waste material. SCG is usually used as a natural amendment for farming purposes [5], but it is also used to produce biodiesel, biogas, bioethanol, and various extraction products for cosmetics and medical industry [6,7]. From the aspect of circular economy, SCG is a cheap raw material that can be used as a filler in composite materials [8].

Unsaturated polyester resins (UPRs) are a significant group of thermosetting polymers. Global production of UPRs is around 12 million metric tons a year, which makes them one of the mass-produced polymers. UPRs can have a wide range of properties due to the large number of materials that are used for their synthesis. They can be used as pure resins or reinforced with suitable fillers. Pure resins are used in production of decorative products, in polymer concrete, as a matrix in composite materials and as a binding agent for various types of coatings [9]. There is an extended supply of fillers that can be used as reinforcement. In this context, the use of natural fibers and particles, such as cotton, flax, or jute, for production of polymer based composite materials is especially appealing [10].

In general, UPRs consist of two main components: unsaturated polyesters (prepolymers) and reactive diluents (RD). In addition to the main components being obtained from petroleum, it has been found that the industrially used reactive diluent styrene poses a potential health risk and has a negative effect on the environment [11]. Constant oil price fluctuations caused by various socio-political events, the gradual depletion of fossil fuels and growing environmental issues have established new trends within the polymer industry. In the last decade, there are great efforts directed towards replacement of petroleum based raw materials with biobased raw materials and transitioning to cleaner technologies [12]. Under these circumstances it is important to find an adequate biobased alternative to styrene. *Cousinet et al.* investigated vinyl levulinate as a potential alternative to styrene, however, obtained UPR showed lower α relaxation temperature, elastic moduli at the rubbery plateau and mechanical properties determined by the three points bending tests [13]. Biobased butanediol dimethacrylate UPR has shown comparable properties to styrene cured UPR, but with poor ductility and resilience.

The use of itaconate based RDs turned out to be a good alternative. Itaconates are less toxic, and volatile compared to styrene without negative environmental effects. However, the mechanical properties of the UPRs with itaconate based RDs still need to be improved [14]. With this regard, to reduce the overall discrepancy in compared to commercial styrene based UPRs, the addition of different reinforcement fillers could be solution of choice.

In this paper a biobased UPR was synthesized as a matrix, with SCG as a biobased filler for a fully biobased composite material. UPR consisted of itaconic acid, succinic acid, propylene glycol with dimethyl itaconate as a RD, all derived from renewable resources. The obtained composite materials were characterized by uniaxial tensile testing, viscosity measurements, FTIR spectroscopy and gel content analysis.

2 Materials and methods

2.1 Materials

The itaconic acid, succinic acid, propylene glycol, hydroquinone and toluene were supplied from Acros Organics. The catalyst, FASCAT 4100 was procured from PMC Organometallix. The initiator, methyl ethyl ketone peroxide (MEKPO), activator, cobalt octoate, and reactive diluent, dimethyl itaconate (DMI) were all supplied by Sigma Aldrich. All chemicals were used as received.

2.2 Synthesis

UPRs are obtained by the polycondensation reaction between itaconic acid, succinic acid, and propylene glycol, by applying the mole ratio of 1:1:2.1, respectively. Toluene (0.5 wt.% based on monomers) was used to increase the rate of water removal. Hydroquinone (150 ppm) was added as a free radical scavenger and the components were mixed. The reaction was carried out in a three-neck round-bottom flask equipped with a stirrer, Dean-Stark and thermometer, and under nitrogen atmosphere. The temperature range was from 110 to 190°C and was raised by 10°C every hour. The reaction was carried out until the acid value reached 35. Obtained prepolymer was cooled to 90°C and diluted in dimethyl itaconate (30% w/w with respect to the prepolymer) which was used as a reactive diluent.

The prepared UPRs were mixed with MEKPO (2.5% w/w) and cobalt octoate (1% w/w), after which SCG was added in different weight ratios (10, 20, 30%). Previously, SCG was sifted through 200 μm sieves and dried in an air oven at 80°C for 5 hours. After homogenization, the mixtures were poured into Teflon molds, which were placed in an air oven for 24 hours at 60°C to cure. Additionally, all the samples were kept at 120°C for 2 hours to harden.

The acid value was defined as the number of milligrams of KOH needed to neutralize 1 g of resin and was measured according to ASTM D465-01. Around 0.5 g of resin was titrated with a KOH equimolar solution of toluene and isopropyl alcohol (0.3 mol/L).

2.3 FTIR spectroscopy

The Fourier Transform Infrared (FTIR) spectra of powdered samples were recorded in transmittance mode for the wavelength range of 600 – 4000 cm^{-1} with a resolution of 4 cm^{-1}, using Nicolet™ iS10 FTIR Spectrometer.

2.4 Viscosity measurements

Viscosities were measured isothermally at 23°C using an Anthon Paar RheoCompass in Peltier plate geometry. The sample (around 5 mL) was loaded in a 20 mm 1°steel cone with a truncation gap of 25 μm. The shear rate was increased stepwise from 0.01 to 1000 s^{-1}, collecting 31 data points to observe any non-Newtonian behavior. At the given shear rate, the shear stress was measured every 2 s. The data were recorded when the shear rate was stabilized with up to 5% tolerance for three consecutive points. Different samples were measured in triplicate and the viscosities were averaged and reported.

2.5 Gel content

The gel content was calculated by extraction in tetrahydrofuran (THF). The cured samples were cut into rectangular shapes, with dimensions of 40 × 10 × 4 mm, and their weight was determined (W_i). Such prepared samples were immersed into THF for 28 days at room temperature. The insoluble fractions, which correspond to the inflated polymer network, were filtered, carefully dried under vacuum, and then measured (W_{sol}). The gel content was calculated as follows:

$$GC\ (\%) = W_{sol} / W_i \times 100$$

2.6 Uniaxial tensile testing

The uniaxial tensile mechanical properties of the investigated UPR samples were evaluated using the Shimadzu Autograph AGS-X servo-hydraulic testing machine, equipped with a 1 kN load cell at ambient temperature according to ASTM D638. Four measurements were performed for each UPR sample at a testing rate of 2 mm/min. The average values of the break stress and break stroke strain, the standard deviations and Young's modulus were determined. Young's modulus was calculated by the software TRAPEZIUM X from the linear part of the stress–strain curve.

3 Results and discussion

SCG composition can vary depending upon the roasting and processing techniques but generally consist of more than 50% carbohydrates that are mainly hemicellulose and cellulose that include their hydrolysis products, while the remaining part comprises lignin, lipids, proteins, minerals, and other substances [15]. Due to the heterogeneous nature of SCG, FTIR spectroscopy was used to determine its chemical structure. The FTIR spectra of SCG (Figure 1) shows a broad characteristic band at 3325 cm^{-1} which corresponds to – OH stretching vibrations. The double peaks at 2924 and 2850 cm^{-1} are related to C–H stretching. The characteristic band at 1656 cm^{-1} is attributed to coupling the C=O aldehyde and the C=C axial deformation, while the characteristic band at 1061 cm^{-1} represents conjugated C–O–C and O–C–C bonds [16].

The chemical structure of both uncured UPR (u-UPR) and cured UPR (c-UPR) was analyzed by FTIR spectroscopy and corresponding spectra are shown in Figure 1. The spectra of u-UPR and c-UPR both show a band at 1720 cm^{-1} that corresponds to C=O stretching, that derives from itaconic acid and succinic acid, and indicates an ester bond forming between carboxyl and hydroxyl groups [17, 18]. Band at 1640 cm^{-1} originates from C=C bonds and decreases in intensity after curing but doesn't completely disappear, which indicated that not all bonds reacted during crosslinking. Bands at 2940 cm^{-1} and 2980 cm^{-1} found in both spectra correspond to C–H stretching vibrations. Bands from 1300 cm^{-1} to 1000 cm^{-1} are related to C–O–C and C–O vibrations of ester bonds.

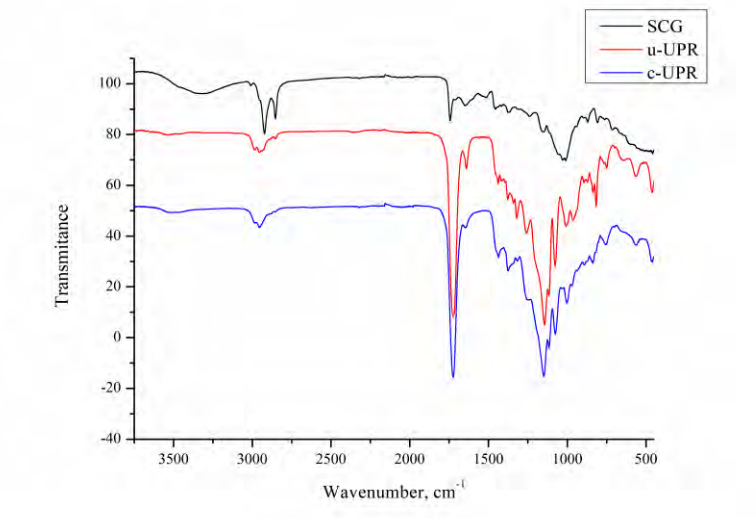

Figure 1. FTIR spectra of SCG, u-UPR and c-UPR.

The FTIR spectra of obtained composite material with different weight ratios of SCG are shown in Figure 2. Wide band at around 3300 cm^{-1} correspond to – OH stretching vibrations and it increases with increasing SCG ratio.

All the bands present in neat SCG spectra are also present in spectra of obtained composite materials. It is important to note that there are no strong interactions between these two materials. Based on these results it could be expected that incorporation of SCG will decrease mechanical properties of composite materials due to poor interaction between matrix and filler particles.

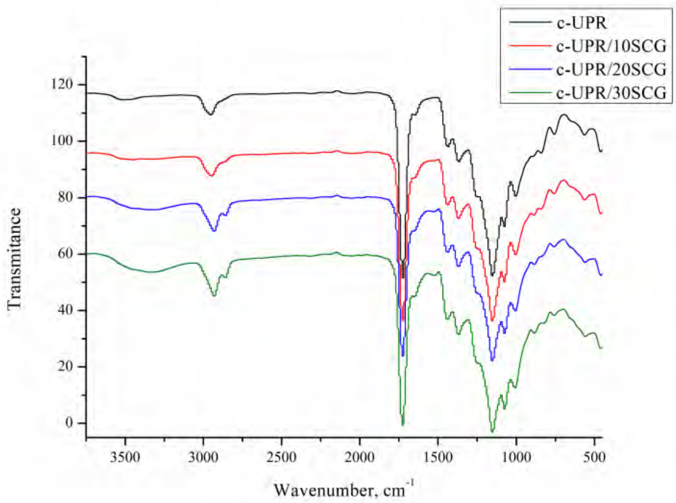

Figure 2. FTIR spectra of obtained composite materials.

The gel content analysis was used to assess the degree of crosslinking. Namely, unreacted monomers, oligomers and non-crosslinked polymer chains would dissolve in THF and leak from the samples. Thus, lower value of gel content indicates lower degree of crosslinking. This property is important for evaluation because it governs mechanical properties of cured resins. The obtained values of gel content are given in Table 1. There is a clear trend indicating that with increasing SCG ratio, the degree of crosslinking is decreasing. This trend showed that the presence of SCG interferes with the polymerization reaction, therefore resulting in lower crosslinking density. Considering the complex structure of waste coffee, several different scenarios may take place to interfere the radical polymerization. Numerous studies found various antioxidants substances in SCG, such as polyphenols, flavonoids, etc. [19, 20]. Most likely these components act as free radical scavengers and inhibit free radical polymerization leading to lower degree of crosslinking.

Table 1. Results of gel content analysis (GC) and uniaxial tensile testing.

Samples	GC, %	Young's modulus, MPa	Break stress, MPa	Break strain, %
c-UPR	98.58	304.55	22.13	13.40
c-UPR/10SCG	96.45	150.23	10.96	11.09
c-UPR/20SCG	92.08	75.06	7.18	10.76
c-UPR/30SCG	91.46	47.68	4.09	8.69

The mechanical properties of obtained composite materials as well as of neat UPR were examined by uniaxial tensile test. The obtained results are summarized in Table 1 and Figure 3. The highest break stress was obtained for c-UPR, while this value deceased with increasing SCG content. Samples c-UPR/20SCG and c-UPR/30SCG had similar degrees of crosslinking, but there is a significant weakening of mechanical properties with increase in SCG content. This could be explained by poor interactions between resin and SCG. Basically, in this system two main factors affect mechanical properties – i.e., degree of crosslinking and interactions between filler and matrix. FTIR analysis showed weak interactions between SCG and UPR. Also, increase in SCG content led to the decrease in degree of crosslinking. Due to this increase in SCG content led to the significant decrease in mechanical properties. Sample with 10 wt.% of SCG (c-UPR/10SCG) showed satisfactory properties to be used in some application while samples with higher content of SCG could not be commercially used. To use higher amounts of SCG it is necessary to modify surface of SCG in order to both increase interaction between SCG and resin, and to decrease inhibition caused by SCG.

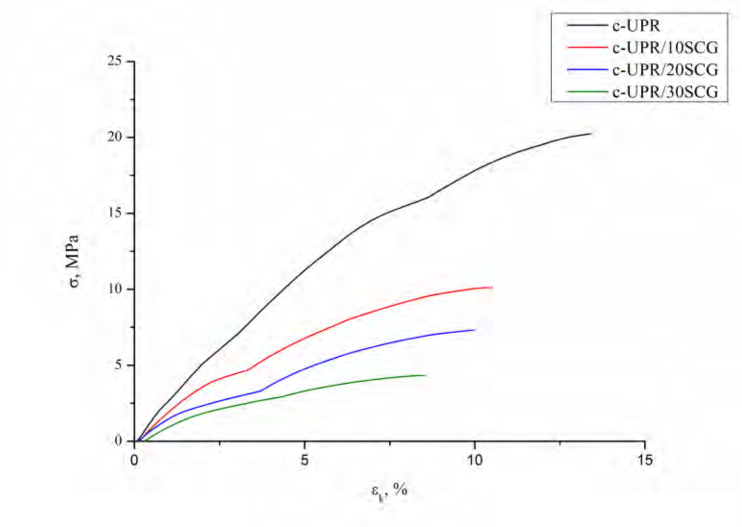

Figure 3. Stress – strain curve.

Shear rate dependance of viscosity of examined composite materials measured at 23°C is shown in Figure 4. Uncured UPR acts as Newtonian liquid showing no dependency on applied shear rate. Within the investigated shear rates, the UPRs with SCG added showed increase in apparent viscosity when compared with u-UPR. With increasing shear rate the slight viscosity decrease could be observed for u-UPR/10SCG and u-UPR/20 SCG. While viscosity of the sample with 30 wt.% of SCG (c-UPR/30SCG) plummeted in the shear rate range from 50 to 300 s^{-1}. This drop in viscosity found in c-UPR/30SCG can be assigned to clusters of SCG that further indicate that there are poor interactions between UPR and SCG.

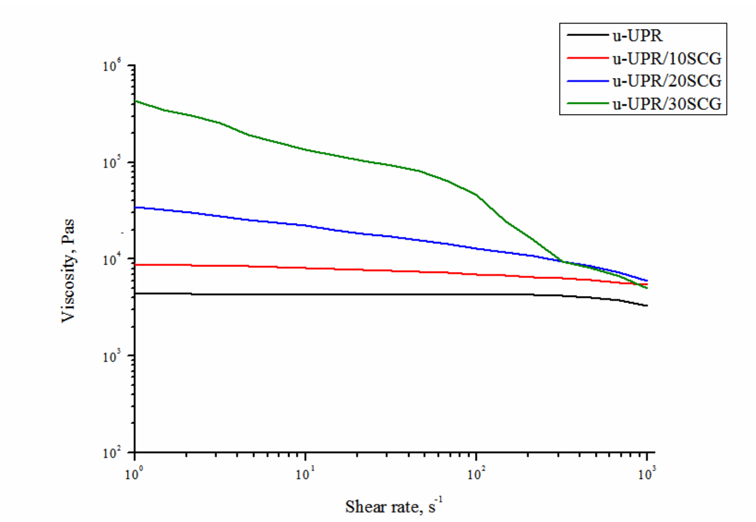

Figure 4. Viscosity as a function of the shear rate of the examined composite materials.

4 Conclusion

In this paper we have investigated the potential use of SCG as a filler to produce a fully biobased composite material. As the matrix, a fully biobased UPR was prepared from itaconic acid, succinic acid, and propylene glycol. Several methods were applied for characterization. FTIR spectroscopy did not reveal any significant interactions between SCG and UPR. Furthermore, the results of gel content analysis indicated that SCG acts as an inhibitor and lowers the degree of crosslinking. As a result, obtained samples showed poor mechanical properties. Only the sample with 10 wt.% of SCG could be commercially used while samples with higher coffee content exhibited unsatisfactory mechanical properties for commercial usage. Viscosity measurements of composite materials further

favored the sample with 10 wt.% of SCG, since materials with high viscosity aren't commercially applicable.
To increase the compatibility between two main system components it is necessary to functionalize the surface of SCG. This could be the way to prevent the inhibition and therefore improve the interactions between SCG and UPR. Since SCG is categorized as a natural filler obtained from lignocellulose raw materials, it has hydrophilic properties. Increasing the compatibility between two main system components can be achieved by lowering the hydrophilicity of SCG. To achieve this objective several approaches can be used, such as selective surface modification of SCG, modification of the UPR matrix by additives and using compatibilizers to provide increased interactions among composite components.

5 Nomenclature

DMI – dimethyl itaconate
MEKPO – methyl ethyl ketone peroxide
RD – reactive diluent
SCG – spent coffee grounds
THF – tetrahydrofuran
UPR – unsaturated polyester resin

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