Non destructive analysis of bitumen-lignin composites by using multispectral optical methods

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Introduction

Recent Green Deal European strategy requires involvement of natural raw materials in products. Lignin, a by product from wood processing, is widely used as carbon source and biodegradable material for different applications such as road coating. Investigation of structure and chemical properties of the lignin modified products by standard methods (SEM, TEM, XPS, etc.) meet some difficulties due to probe preparation and sample destroying during investigation. In this situation, combination of optical methods, such as UV-Vis spectroscopy, photoluminescence (PL) spectroscopy and FTIR method could provide information on chemical structure, phase transitions and degradation of the lignin-based materials.

Experimental

Bitumen-lignin composites were prepared by mixing of B70/100-grade bitumen with sulfolignin. Ageing of Bitumen and Bitumen-lignin composites have been performed in a climate chamber (135 ^{0}C, 10 atm, 70% RH, 1 month). FTIR spectroscopy was performed in the absorbance mode in a range of 4000-380 cm^{-1} (Brucker Alfa II spectrometer). Photoluminescence measurements were performed with a fiber optic spectrometer (Ocean Optics HR4000) in range 350-800 nm. The excitation source was a UV LED (310 nm, FWHM 10 nm, 3 mW output power).

Results and discussion

FTIR spectra of bitumen, and bitumen-lignin are shown in Fig. 1a. Bitumen showed characteristic peaks, located at 3300, 2900, 2800 and 2000-1000 cm^{-1}, corresponding to O-H, sp3, sp2 hybridization and C-H, C=C and C-O groups. Adding lignin to bitumen changed FTIR spectra drastically. New peaks appeared at 3300 cm-1, 1689, 1650, 1134, 1021 corresponding to O-H, C=O, C=C and C-O vibrations in polyphenolic molecules of lignin.

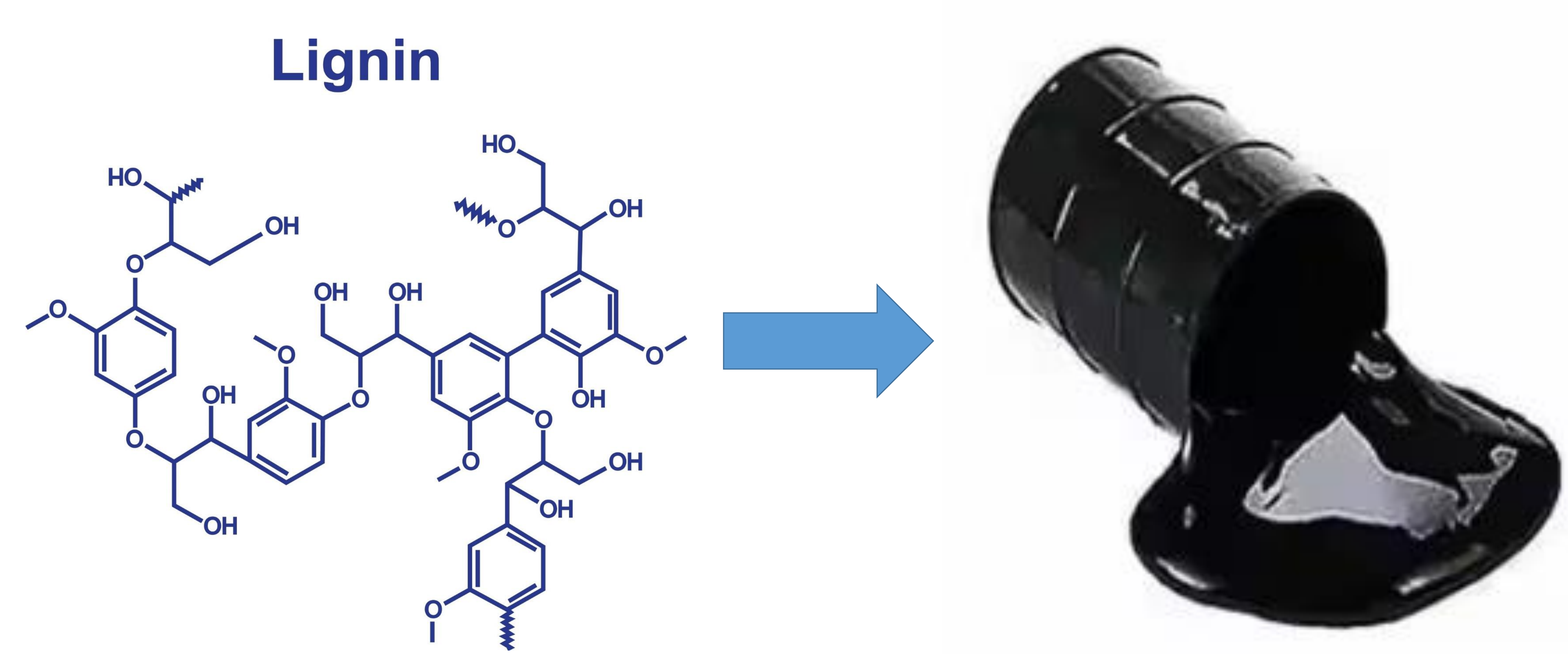
Normalized photoluminescence spectra of bitumen and bitumen-lignin composites are shown in Figure 1b. It is worth to note that lignin quenched photoluminescence of the bitumen samples as lignin absorbs light in UV-Vis range. Therefore, the peak shift of PL maximum in bitumen-lignin spectra could be explained by lignin absorbance.

FTIR spectra of aged samples of bitumen, and lignin-bitumen are shown in Fig 2a-c. Aging process changed the composition of bitumen. The FTIR of aged bitumen showed additional peaks at 3200, 1713, 1600, 1456, 1383, 1025 cm^{-1}. The observed peaks are related to O-H, C=O, C=C, C-O vibrations. The ratio between sp3 and sp2 peak intensities changed after ageing. It is supposed that the found change of the FTIR spectrum are related of the compounds, formed as products of oxidation reaction into bitumen. Opposite to that, FTIR spectra of bitumen-lignin composites showed no drastic changes after artificial ageing. The only increase of the peak at 3200 cm^{-1} could be explained by O-H vibration of the water molecules, adsorbed into bitumen-lignin composites.

Ageing process provoked changes in emission of bitumen and bitumen-lignin composites. The PL maximum of the aged bitumen shifted from 580 nm to 600 nm. The bitumen-lignin PL spectra showed no peak shift. For low concentration of lignin (10%) it was observed an intensity increase in the range of 580-600 nm, whereas no change of the spectrum was observed for high concentration of lignin (Fig2d.). Comparing the PL changes of bitumen and bitumen-lignin (10%) we can assume that PL changes point to structure changes of the bitumen induced by artificial ageing. Thus, a partial oxidation of bitumen took place at low concentration of lignin.

Conclusions

Developed methodology, based on UV-VIS and IR methods showed good results in analysis of bitumen-lignin composites with different lignin concentration. Combination of FTIR and PL allows discriminate and explain degradation effects in bitumen. This methodology can be applied for other objects like polymeric composites and their degradation.



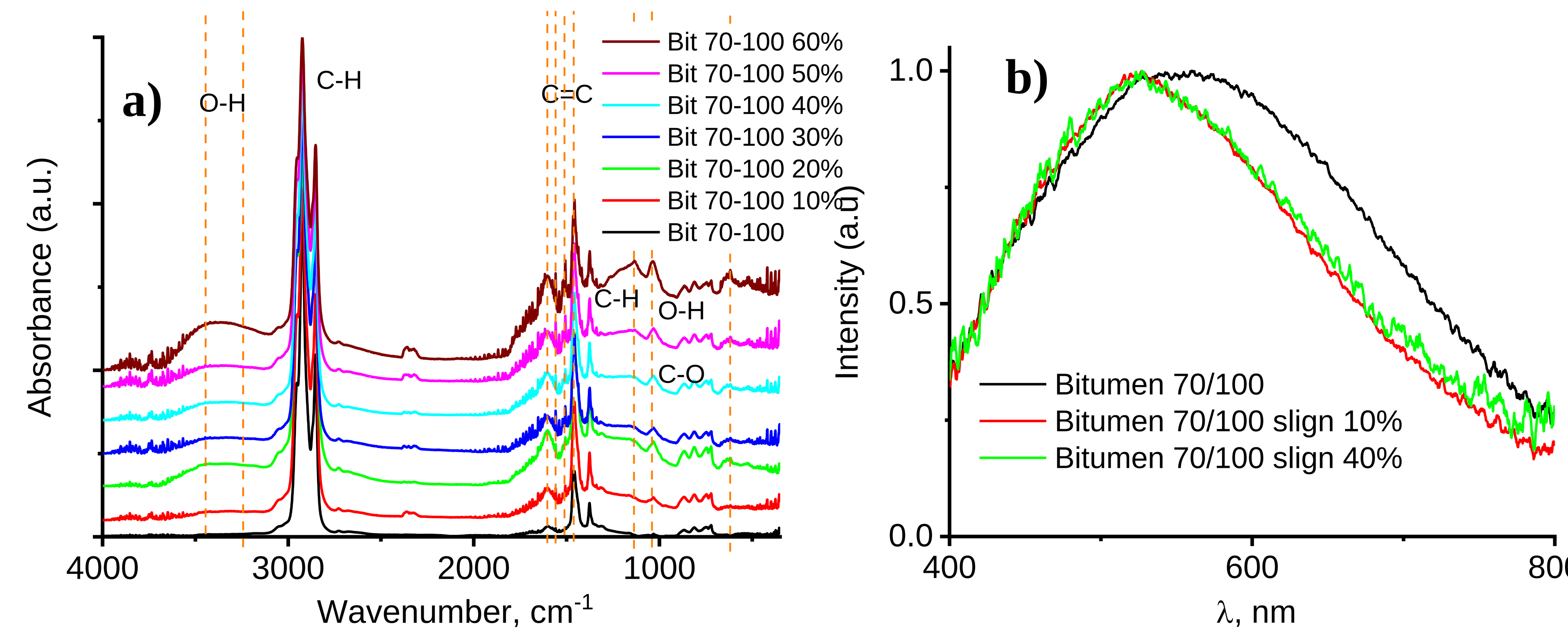


Figure 1. FTIR spectra of bitumen and bitumen lignin composites (a) and PL spectra of bitumen and bitumen lignin composites (b)

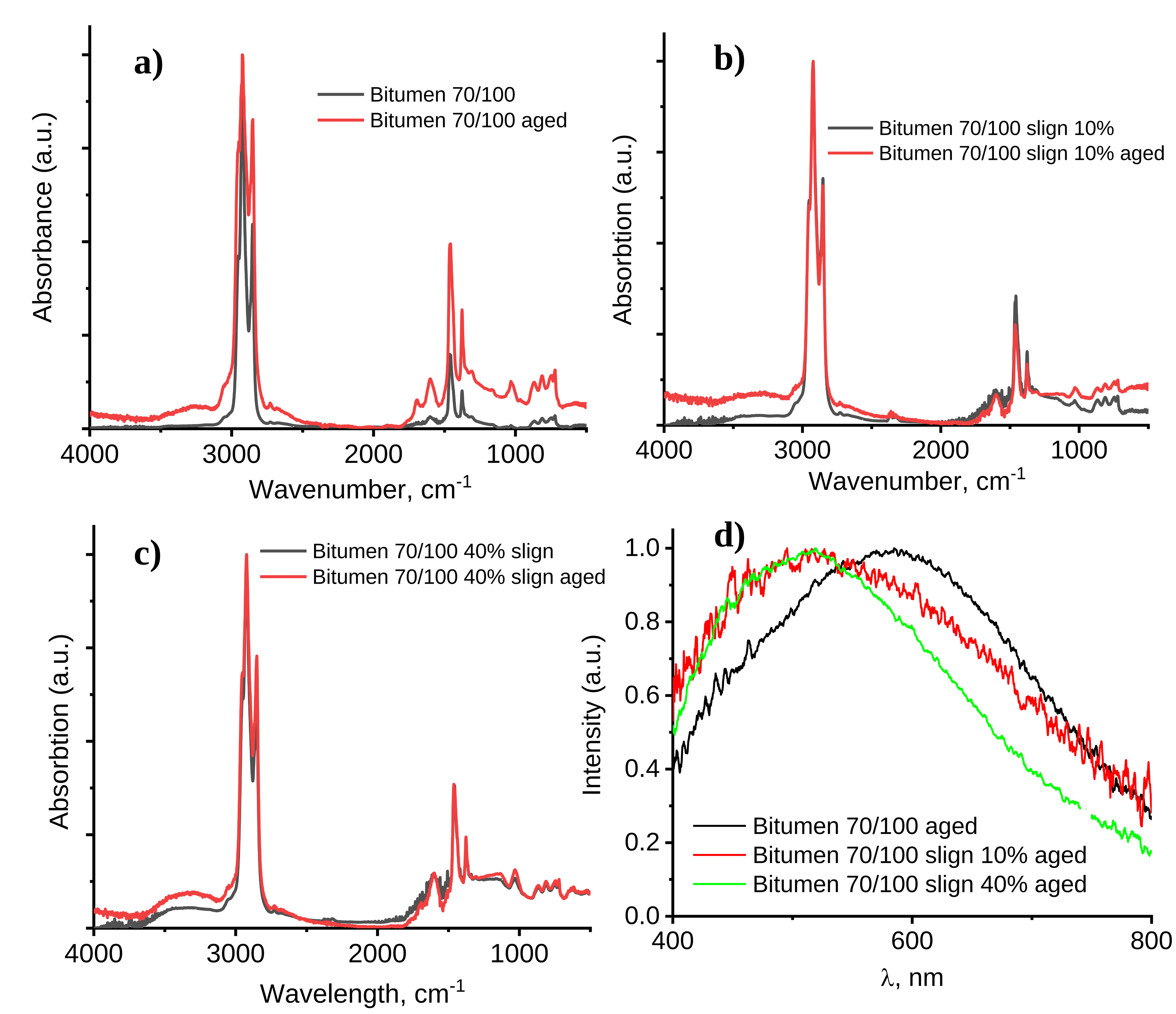


Figure 2: FTIR spectra of bitumen and bitumen lignin composites before and after ageing (a) pure bitumen, b) bitumen-lignin 10% , c) bitumen-lignin 40% , d) PL spectra of bitumen and bitumen-lignin composites after ageing

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