VANILLIN ACRYLATE-BASED PHOTOCROSS-LINKED POLYMERS: SYNTHESIS AND INVESTIGATION OF PROPERTIES

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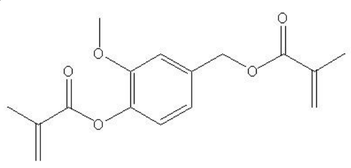
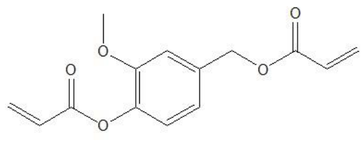
THE AIM

The aim of this work was to investigate the kinetics of free-radical photopolymerization of vanillin diacrylate and vanillin dimethacrylate using ethyl(2,4,6-trimethylbenzoyl) phenylphosphinate as photoinitiator, as well as rheological properties of the photocross-linked polymers by real-time photorheometry. The influence of the resin composition to UV/VIS curing time and rigidity of the resulted polymers and their thermal properties was investigated.

PHOTOCROSS-LINKING*

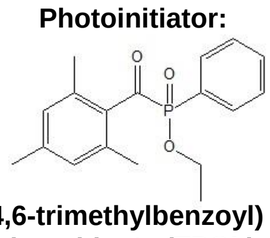


Vanillin dimethacrylate (VDMA)



Vanillin diacrylate (VDA)

Photoinitiator:



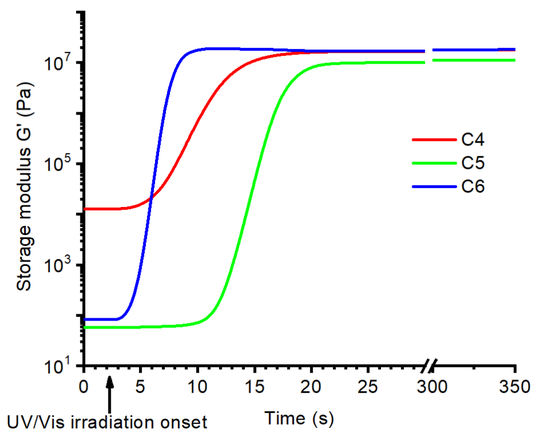
Ethyl(2,4,6-trimethylbenzoyl) phenyl phosphinate (TPOL)

Resin	Vanillin derivative	Amount of TPOL, mol.%	Solvent
C1	VDA	1	-
C2	VDA	1	DCM
C3	VDMA	1	DCM
C4	VDA	3	-
C5	VDA	3	DCM
C6	VDMA	3	DCM
C7	VDA	5	-
C8	VDA	5	DCM
C9	VDMA	5	DCM

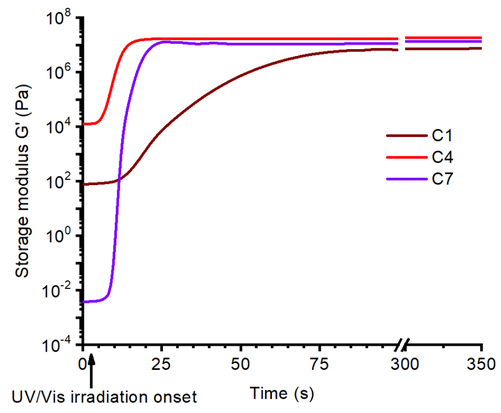
- **Photocross-linking was performed using 500 W Helios Italquartz lamp (250-450 nm, 310 mW·cm^{-2}).**

(DCM – dichloromethane)

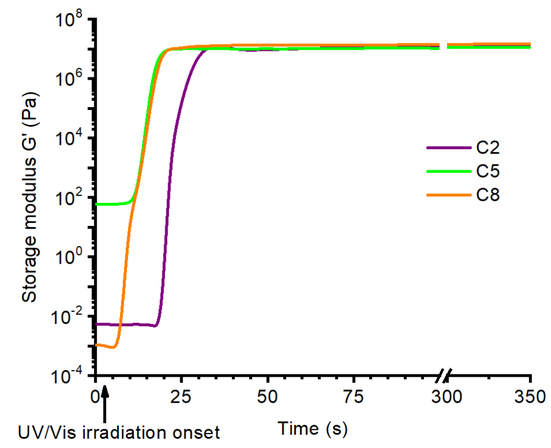
DEPENDENCE OF STORAGE MODULUS G' ON RESIN COMPOSITION



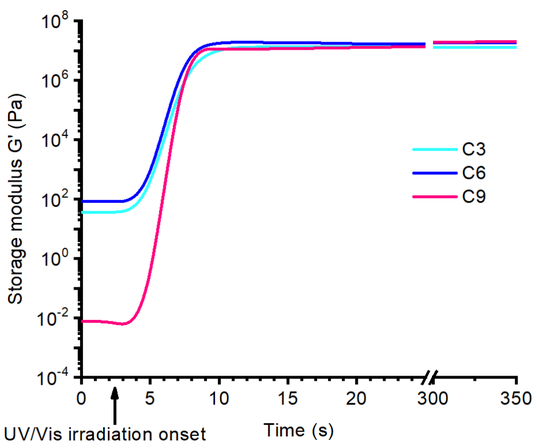
Dependencies of storage modulus G' of the resins C4, C5 and C6 with 3 mol.% of photoinitiator on irradiation time



Dependencies of storage modulus G' of the VDA-based resins C1, C4 and C7 with different amount of photoinitiator on irradiation time



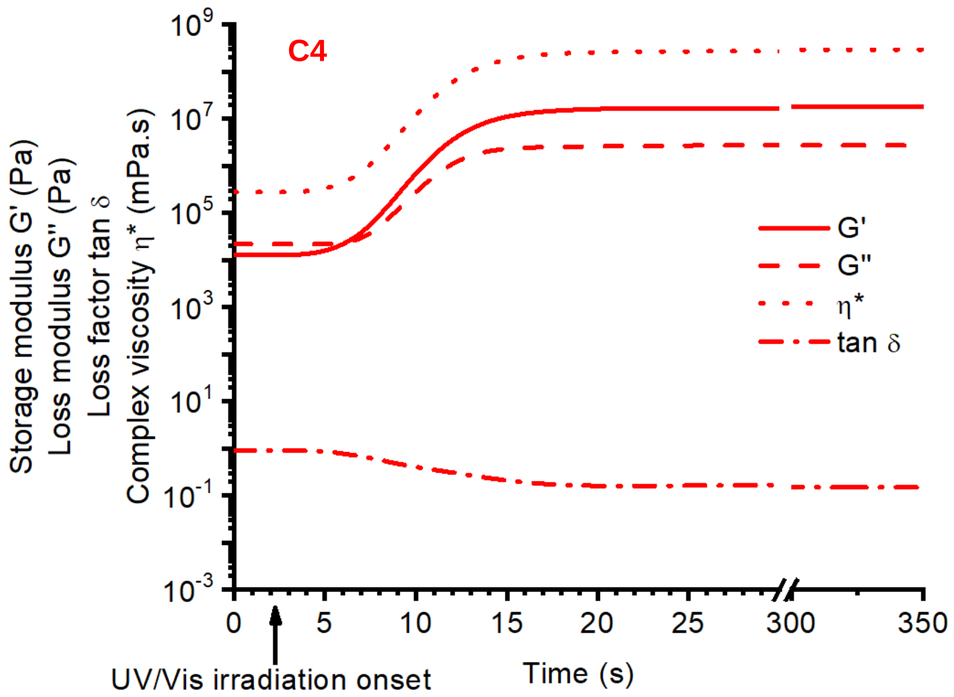
Dependencies of storage modulus G' of the VDA-based resins C2, C5 and C8 with different amount of photoinitiator on irradiation time



Dependencies of storage modulus G' of the VDMA-based resins C3, C6 and C9 with different amount of photoinitiator on irradiation time

RHEOLOGICAL CHARACTERISTICS OF RESINS

Resin	Storage modulus G', MPa	Complex viscosity η*, MPa·s	Loss modulus G'', MPa	Gel point* t_{gel}, s
C1	7.35	0.154	6.34	10
C2	13.40	0.217	2.35	20
C3	13.00	0.208	1.65	6
C4	18.10	0.290	2.70	6
C5	11.30	0.180	1.64	12
C6	18.20	0.290	2.94	5
C7	13.30	0.230	5.78	10
C8	14.50	0.230	2.02	14
C9	19.80	0.319	3.36	6



***- calculated from the UV/VIS irradiation onset.**

Dependencies of storage modulus G', loss modulus G'', loss factor tan δ, and complex viscosity η* of resin C4 on irradiation time

THERMAL PROPERTIES

Glass transition temperature (T_g) of the photocross-linked polymers was estimated by differential scanning calorimetry (DSC).

Thermal decomposition temperature at the weight loss of 10% ($T_{dec.-10\%}$) was determined by thermogravimetrical analysis (TGA).

Polymer	$T_{dec-10\%}$, ˚C	T_g, ˚C
C4	350	87
C5	330	63
C6	340	86

CONCLUSIONS

- **The curing time was the shortest and the most rigid polymers were obtained when vanillin dimethacrylate or vanillin diacrylate and ethylphenyl(2,4,6-trimethylbenzoyl) phosphinate were used in the compositions without dichloromethane.**
- **The shortest reaction time was obtained when 3 mol.% of the ethylphenyl(2,4,6-trimethylbenzoyl) phosphinate were used in the compositions.**
- **Addition of dichloromethane into the resins resulted in less rigid polymers and longer UV curing time.**
- **Vanillin diacrylate-besed polymer without any solvent performed higher thermal characteristic values.**

ACKNOWLEDGEMENT

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