Hydrophobization of lignocellulosic materials part II – chemical modification

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Table 1 . Overview of modifications of cellulosic materials by esterification. Thermomechanical pulp (TMP), chemi-thermomechanical pulp (CTMP), microcrystalline cellulose (MCC), cellulose nanocrystal (CNC), cellulose nanostructures (CNS), bacterial nanocellulose (BNC), vegetal cellulose (VC), trifluoroacetic anhydride (TFAA), microfibrillated cellulose (MFC), cellulose nanowhiskers (CNW).

Cellulose source/ substrate	Type of cellulose	Esterifying agent	Degree of substitution (DS)	Contact angle (°)	Ref.
Surface					
Unbleached spruce TMP	Fibers	AcOH	0.05-0.62*		(Paulsson et al. 1994)
Bleached spruce TMP			0.0-0.67*		
Bleached aspen CTMP			0.03-0.29*		
Tunicin	MCC	AcOH /Ac_2O	Homogeneous: 2.81 Heterogeneous: 2.3		(Sassi and Chanzy 1995)
Cellulosic waste materials	CNC	Ac_2O	2.18	76.54	(Abraham et al. 2016)
Avicel	CNC	Ac_2O	0.32-1.98		(Wu et al. 2018)
MCC from bleached kraft pulp of eucaliptus	CNS	AcOH /Ac_2O	Hydrolysis of MCC with H_2SO_4: 1.5-2.1 Hydrolysis of MCC with HCl: 1.4-1.8		(Barbosa et al. 2019)
Avicel	CNC	AcOH /Ac_2O	0.18, 0.42		(Xu et al. 2020)
MCC from wood pulp	CNC	Ac_2O	0.18, 0.34		(Ávila Ramírez et al. 2017)
Bleached beech dissolving pulp, non-bleached beech	Fibers	N-acetylimidazole	0.17	Beached: 50 Unbleached: 73	(Beaumont et al. 2020)

organosolv pulp					
Avicel, sugar cane bagasse	MCC, fibers	Octadecanoyl chloride		76-88	(Pasquini et al. 2006)
		Dodecanoyl chloride		76-83	
Cotton fabric	Fibers	Chloroacetyl chloride		110	(Jantas and Górna 2006)
Never-dried bleached beech sulfite dissolving pulp	Fibers	N-acetylimidazole	0.17	52	(Beaumont et al. 2021b)
		N-isobutylimidazole	0.10	61	
		N-succinylimidazole	0.25		(Beaumont et al. 2021c)
BNC from Nata-de-coco	Homogeni-zed BNC	AcOH	0.0051**	75***	(Lee et al. 2011)
		Hexanoic acid	0.0029**	92***	
		Dodecanoic acid	0.0027**	133***	
BNC	Homogeni-zed BNC	AcOH /Ac_2O	0-0.73	$DS_{0.25}$= 81 $DS_{0.63}$= 88	(Ávila Ramírez et al. 2016)
BNC, α-cellulose (VC)	BNC, fibers	Ac_2O	BNC: 0.17 VC: 0.10	BNC: 79 VC: 80	(Tomé et al. 2011)
		Butyric anhydride	BNC: 0.24 VC: 0.22	BNC: 95 VC: 102	
		Hexanoic anhydride	BNC: 0.13 VC: 0.12	BNC: 104 VC: 105	
		ASA	BNC: 0.04 VC: 0.06	BNC: 97 VC: 95	
		Hexanoyl chloride	BNC: 0.17 VC: 0.02	BNC: 109 VC: 113	
Avicel	MCC	Trimethylsilyl-diazomethane			(Müller et al. 2010)
Filter paper, tunicate	Fibers, nanofibers	AcOH/ TFAA Ac_2O/TFAA	AcOH/TFAA Bulk: 0.082 Surface: 1.09	Filter paper> 90	(Yuan et al. 2005)
Kraft pulp of Norway spruce	MFC	TFAA/AcOH		66.6-89.7	(Rodionova et al. 2013)
		TFAA/Ac_2O		83.3-96.9	
Pine cellulose	Cellulose particles	Palmitoyl chloride	0.01-0.14	98-109	(David et al. 2019)
Never-dried hardwood pulp	MFC	Palmitoyl chloride	0.04-2.36		(Fumagalli et al. 2013a)
Cotton linters	CNC	Palmitoyl chloride	0.07-2.50		(Fumagalli et al. 2013b)
Never-dried hardwood pulp	MFC	Palmitoyl chloride	0.98		(Fumagalli et al. 2015)
		Decanoyl chloride	1.01		
		Sebacoyl chloride	0.15		
		(2-dodecen-1-yl) succinic anhydride	0.13		

Avicel	CNW	Vinyl acetate			(Çetin et al. 2009)
Prehydroly-zed kraft rayon-grade dissolving eucalyptus dry lap wood pulp	CNC	Canola oil fatty acid methyl esters		68	(Wei et al. 2017)
Cellulose powder	Nanofibers	Hexanoyl chloride	0.58		(Huang et al. 2012)
Cellulose powder	Nanofibers	Pentafluoro-benzoyl chloride	Reaction in toluene: 0.20 Reaction in DMF: 0.57	Reaction in toluene: 133 Reaction in DMF: 102.7	(Rao et al. 2015)
Totally chlorine free-bleached beech sulfite dissolving pulp	Fibers	N-acetylimidazole	0.49	~134	(Beaumont et al. 2021a)
Bulk					
	CNC	Ac_2O	2.16		(Li et al. 2019)
Viscose rayon (staple fiber)	Fibers	AcOH	3		(Shimizu and Hayashi 1988)
Bleached sulfite pulp			2.44 Mercerized before modification: ~3		
Cotton linter			2.10 Mercerized before modification: ~3		
Highly purified bleached sulfite pulp	Fibers	Undecylenic acid	0.47, 1.11		(Jandura et al. 2000)
		Undecanoic acid	0.31, 0.59		
		Oleic acid	0.08, 0.14		
		Stearic acid	0.12, 0.19		
Kenaf bast fibers	Fibers	Ac_2O	1.16	113	(Jonoobi et al. 2010)
	Nanofibers		1.07	115	
Wheat fibers	Fibers/ nanofibers	Succinic anhydride	0.67		(Sehaqui et al. 2017)
		Maleic anhydride	0.15		
		Phthalic anhydride	0.17		
Posidonia oceanica	Fibers	Succinic anhydride	0-2.24		(Chadlia and Farouk 2011)
		Maleic anhydride	0.04-1.59		
		Phthalic anhydride	0.12-1.5		
MCC	MCC	Stearoyl chloride	2.95	158	(Geissler et al. 2013)
MCC	MCC	Vinyl laureate	1.47-2.74	96.3-120.7	(Wen et al. 2017)

*Calculated based on the formula from (Fumagalli et al. 2013b)

**Calculated based on the calculations of (Cunha et al. 2014) and using the specific surface area from (Lee and Bismarck 2012).

***Advancing contact angles

Table 2. Overview of modifications of cellulosic materials by silylation. Trichloromethylsilane (TCMS), microcrystalline cellulose (MCC), cellulose nanocrystal (CNC), bacterial nanocellulose (BNC), methyltrimethoxysilane (MTMS), hexamethyldisilazane (HMDS), n-dodecyltrimethoxysilane (n-DDTS), and perfluorooctyltriethoxysilane (PFTS), butyltrichlorosilane (BTCS), 3,3,3-trifluoropropyl trimethoxysilane (TFPS) and 1H,1H,2H,2H-perfluorooctyl trimethoxysilane (PFOTMS), cellulose nanofiber (CNF), microfibrillated cellulose (MFC), (3-aminopropyl)-trimethoxysilane (APTES), chlorodimethyl isopropylsilane (CDMIPS).

Cellulose source	Type of cellulose	Silylating agent	DS	CA (°)	Ref.
Surface					
Whatman filter paper	Fibers	TCMS		125-136	(Cunha et al. 2010)
Whatman filter paper	Fibers	MTMS, HMDS, *n*-DDTS, PFTS		124-146	(Yu et al. 2019)
MCC	MCC			94-133	
Viscose rayon	Fibers			90-147	
Regenerated cellulose	Fibers			102-120	
CNC	CNC			123-136	
BNC	BNC			0-136	
Cotton	Fibers	TCMS	0.10	101	(Jarrah et al. 2018)
		Dichlorodimethyl silane	0.29	97	
		BTCS	0.08	110	
		Trichloro(3,3,3-trifluoropropyl)silane	0.13	59	
Paper from northern bleached softwood Kraft fibers mixed with 15% bleached softwood/85% bleached hardwood fibers	Fibers	TCMS		~150	(Tang et al. 2016)
		BTCS		~125	
		Dodecyltrichlorosilane		~130	
		Octadecyltrichlorosilane		~140	
Avicel	MCC, fibers	TFPS, PFOTMS		TFPS:115 PFOTMS:125	(Ly et al. 2010)
Whatman filter paper				TFPS:116 PFOTMS:129	
Chlorine free cellulose pulp (spruce (60–70%) and pine fibers (30–40%))	CNF	TCMS	Total: 1-6% Surface: 21-97%	Dense films: 150-160 Porous films: 157-163	(Orsolini et al. 2018)
Recycled box board	CNF	50% MTMS / 50% HDTMS	6.27 at-%	107.2	(Laitinen et al. 2017)
Recycled milk container			4.80 at-%	129.4	
Fluting board			5.00 at-%	159.0	
Bleached birch kraft pulp sheets			4.47 at-%	142.9	
Canola straw fibers	CNF	HDTMS		121.1-138.9	(Rafieian et al. 2018)
Unbleached long fiber of *Pinus elliottii*	CNF	MTMS		Bulk: 110.24, 113.16 Surface: 119.85	(Lazzari et al. 2017)

Pulp powder from oat straw	CNF	MTMS	0-38 wt% Si	105-136	(Zhang et al. 2014)
Recycled cellulose fibers from paper waste	Fibers	MTMS		External: 153.5 Internal: 150.8	(Feng et al. 2015)
Recycled cellulose fibers from paper waste, cotton fibers	Fibers	MTMS		130.3-142.8	(Cheng et al. 2017)
Never-dried ECF-bleached birch kraft pulp	CNF	(tridecafluoro-1,1,2,2-tetrahydrooctyl) trichlorosilane		160	(Jin et al. 2011)
Nata de coco (BNC)	CNF	Trimethylchlorosilane/ triethylamine	0.075-0.132	137.1-146.5	(Sai et al. 2015)
Bleached and unbleached *Pinus radiata* pulp fibers	MFC	HMDS		Bleached: 89-101 Unbleached: 88-97	(Chinga-Carrasco et al. 2012)
Never dried birch pulp	CNF	HMDS	0.05-0.3*	~70	(Peresin et al. 2017)
		APTES	0.5-0.7*	~60	
Sugar beet pulp pulp	MFC	CDMIPS	~0.04-0.36		(Goussé et al. 2004)
Bleached softwood sulfite pulp	MFC	CDMIPS	0.03-0.95	117-146	(Andresen et al. 2006)
Cellulose pulp powder from oat straw	CNF	MTMS	17.4 wt%	110-147	(Zhang et al. 2015)
BNC	BNC	MTMS	0.41-5.90		(Kono et al. 2020)
Cellulose II gel		3-azidopropyl triethoxysilane	1.2 mmol Si/g		(Beaumont et al. 2018)
Never-dried beech sulfite dissolving pulp	CNF		0.52 mmol Si/g		
Carboxymethylated pulp	FIber		0.23 mmol Si/g		

*Surface values determined by XPS

Table 3. Gas permeances of lignocellulosic and plastic materials Low-density polyethylene (LDPE), high-density polyethylene (HDPE), polyethylene terephthalate (PET), cellulose nanofiber (CNF), TEMPO-oxidized cellulose nanofibril (TOCNF), polyethylene (PE), polystyrene (PS), water vapor transmission rate (WVTR), microfibrillated cellulose (MFC), polypropylene (PP).

Material	**O_2 (mL×cm/(m^2× 24hr×atm))**	**Original value (unit)**	**Conversion factor**	**Ref.**
LDPE	16.6 [b]	16.6 mL(STP) cm/(m^2 24 h × atm)	1	(Wang et al. 1998)
LDPE [a]	12.0 [b]	12.0 mL(STP) cm/(m^2 24 h × atm)	1	
HDPE	2.6 [b]	2.6 mL(STP) cm/(m^2 24 h × atm)	1	
Microfibrillated CNF films	>10 [c]	>1000 mL(STP) μm/(m^{2}day× kPa)	10^{-4} (cm/μm)/0.00986923 (atm/kPa) = 0.01	(Fukuzumi et al. 2011)
Cellophane	5×10^{-4} [c]	0.05 mL(STP) μm/(m^{2}day× kPa)	10^{-4} (cm/μm)/0.00986923 (atm/kPa) = 0.01	
Tunicate TOCNF	>10 [c]	>1000 mL(STP) μm/(m^{2}day× kPa)	10^{-4} (cm/μm)/0.00986923 (atm/kPa) = 0.01	
Wood-TOCNF	6×10^{-5} [c]	0.006 mL(STP) μm/(m^{2}day× kPa)	10^{-4} (cm/μm)/0.00986923 (atm/kPa) = 0.01	
PET, Nylon, PE, PS	<1 [d]	<2.88×10^{-13} mL(STP) cm/(cm^{2}s×Pa)	1/(10^{-4}m^2/cm^2 ×1/3600day/s × 9.9 × 10^{-6} atm/Pa) = 3.6 × 10^{12}	(Guisheng et al. 1995)
Material	**Water (WVTR, mg×cm/(m^2×24hr))**	**Original value (unit)**	**Conversion factor**	**Ref.**
Cellulose acetate with various compositions	< 988	<5.28 mg/(h×cm^2)	30×10^{-4}cm /((3.86 cm^2 /10^4) × (1/24)day/h)	(Sprockel et al. 1990)
MFC/CNF films	< 130	51-52 g/(m^2 × day)	1000mg/g × 0.0025 cm/μm = 2.5	(Kumar et al. 2014)
PET, PE, PP	<20	<0.2 g × mm/(m^2 × day)	1000 mg/g ×0.1cm/mm = 100	(Bhadha 1999, Keller and Kouzes 2017)

Material	CO_2 (mL×cm/(m^2× 24hr×atm))	Original value (unit)	Conversion factor	Ref.
PET, Nylon, PE, PS	< 3.6 [d]	< 10^{-12} mL × cm/(s ×cm^2 × Pa)	1/(10^{-4}m^2/cm^2 × 1/3600 day/s × 9.9 × 10^{-6} atm/Pa) = 3.6 × 10^{12}	(Guisheng et al. 1995)
Cellulose acetate and ethyl-cellulose	<1.4	< 5 × 10^{-10} cm^3 × cm/(s × cm^2 × cmHg) (barrer)	1/(1/3600 day/s × 10^{-4} m^2/cm^2 × 0.013 atm/cmHg) = 2.8 × 10^9	(Sanaeepur et al. 2015)

[a)] The LDPE in this study had different densities [b)] Gas permeation values detected at 23-25 °C, ambient. [c)] Gas permeation values detected at 23 °C and 0 % relative humidity. [d)] at 30 °C, ambient. The wording "day" is assumed to be 24 hours.

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