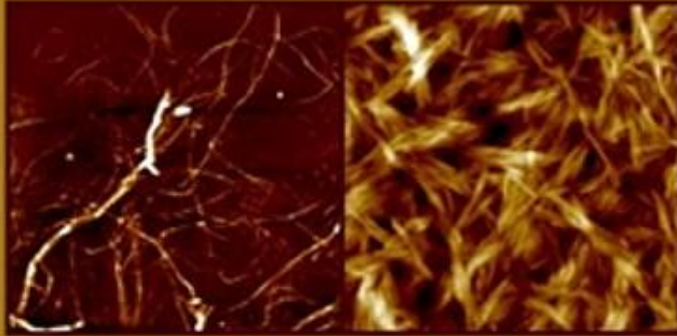


WOODHEAD PUBLISHING SERIES IN COMPOSITES SCIENCE AND ENGINEERING



INDUSTRIAL APPLICATIONS OF NANOCELLULOSE AND ITS NANOCOMPOSITES



Edited by
S.M. SAPUAN,
M.N.F. NORRAHIM, R.A. ILYAS

Industrial Applications of Nanocellulose and Its Nanocomposites

Editors: S.M. Sapuan, M.N.F. Norrahim, R.A. Ilyas

Woodhead Publishing is an imprint of Elsevier
50 Hampshire Street, 5th Floor, Cambridge, MA 02139, United States
The Boulevard, Langford Lane, Kidlington, OX5 1GB, United Kingdom

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ISBN: 978-0-323-89909-3
e-ISBN: 978-0-323-89917-8

For information on all Woodhead publications
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Publisher: Matthew Deans
Acquisitions Editor: Gwen Jones
Editorial Project Manager: Chiara Giglio
Production Project Manager: Surya Narayanan Jayachandran
Cover Designer: Mark Rogers

Typeset by STRAIVE, India



Preface

Industrial Applications of Nanocellulose and Its Nanocomposites provides an extensive, up-to-date overview of this fast-moving area of study from the perspectives of prominent researchers in academic, industrial, and government or private research laboratories. This is an exciting time to be in as, moving beyond scientific curiosity, nanocellulose is starting to hit the marketplace. Nanocellulose is a versatile material that is receiving a lot of attention from scientists in several fields such as automotives, composites, adsorbents, paints, coatings, medical implants, electronics, cosmetics, pulp and paper, tissue engineering, packaging, and aerogels. Current trends show that research related to recent developments of nanocellulose is increasing and covers several aspects including synthesis, surface modification, and improvement of the properties of nanocellulose, bearing in mind the targeted applications.

The objectives of this book are to reflect on recent advancements in the design and fabrication of nanocellulose and to discuss the important requirements for each application, along with the challenges that might arise. This book also includes an overview of the current economic perspectives and safety issues related to nanocellulose. The potential of nanotechnology and nanocomposites in various sectors of research and applications is promising and attracting increasing investment. For this reason, this book will benefit end users such as students, researchers, and industry players. Each chapter explains in detail the important role of nanocellulose, including the advantages and limitations of its specific applications. The book includes commentary from leading industrial and academic experts in the field who present cutting-edge research on advanced materials based on nanocellulose. Improvement features and recommendations are also provided to pave the way to new horizons for nanocellulose, and its applications. Therefore, this book will offer guidance to current, new, and future researchers in nanocellulose to strategize their work to meet the current demands. In terms of commercialization, this book will steer industry players to identify the potential uses of nanocellulose in their products. These same concepts are available elsewhere in the preface. Finally, thoughts on the future directions of nanocellulose-based materials have been included in some chapters.

S.M. Sapuan
M.N.F. Norrrahim
R.A. Ilyas

Table of contents

- Full text access
Front Matter, Copyright, Contributors, Editors' biographies, Preface

- Book chapter Abstract only
1 - Introduction to nanocellulose production from biological waste
R.A. Ilyas, M.R.M. Asyraf, ... Norizan Mohd Nurazzi
Pages 1-37
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
2 - Economic insights into the production of cellulose nanofibrils from oil palm biomass
M.N.F. Norrahim, Mohammed Abdillah Ahmad Farid, ... R.A. Ilyas
Pages 39-48
[Purchase](#) [View abstract](#) ▼









- Book chapter Abstract only
3 - Nanocellulose: Sustainable biomaterial for developing novel adhesives and composites
Zeki Candan, Ayhan Tozluoglu, ... Tufan Salan
Pages 49-137
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
4 - Nanocellulose-based aerogels for various engineering applications
Paulo Henrique Camani, Gabriel Diego Lemes and Derval dos Santos Rosa
Pages 139-153
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
5 - Nanocellulose: Chemistry, preparation, and applications in the food industry
Omar Bashir, Sumira Rashid, ... Beenish
Pages 155-177
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
6 - Nanocellulose nanocomposites in coating materials
Nasmi Herlina Sari, Suteja and R.A. Ilyas
Pages 179-195
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
7 - Nanocellulose as an adsorbent for heavy metals
M.N.F. Norrahim, Noor Azilah Mohd Kasim, ... Keat Khim Ong
Pages 197-211
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
8 - Nanocellulose in sensors
Muhammad Syukri Mohamad Misenan, Z.N. Akhlisah, ... M.N.F. Norrahim
Pages 213-243
[Purchase](#) [View abstract](#) 
- Book chapter Abstract only
9 - An overview of cellulose nanofiber physicochemical characterizations and biological studies in relation to nanosafety concerns
Leo Bey Fen, Jahangir Kamaldin and Hazirah Pengiran
Pages 245-261
[Purchase](#) [View abstract](#) 
- Book chapter Abstract only
10 - Nanocellulose hydrogels
J. Mantovan, J.F. Pereira, ... S. Mali
Pages 263-287
[Purchase](#) [View abstract](#) 
- Book chapter Abstract only
11 - Nanocellulose applications in packaging materials
Tengku Arisyah Tengku Yasim-Anuar, Hidayah Ariffin, ... Mohd Ali Hassan
Pages 289-310
[Purchase](#) [View abstract](#) 
- Book chapter Abstract only
12 - Active biocomposite packaging films: Compatibility of carrageenan with cellulose nanofiber from empty fruit bunches
Nurul Aini Mohd Azman
Pages 311-326
[Purchase](#) [View abstract](#) 
- Book chapter Abstract only
13 - Enhanced thermal stability of cellulose nanocrystals for processing polymer nanocomposites at a high temperature
Khairatun Najwa Mohd Amin
Pages 327-335
[Purchase](#) [View abstract](#) 
- Book chapter Abstract only
14 - Nanocellulose nanocomposites for biomedical applications
Ismail M. Fareez, Ramli M. Zaki and Jasni A. Hawa
Pages 337-352
[Purchase](#) [View abstract](#) 
- Book chapter Abstract only
15 - Nanocellulose biocomposites in specialty papermaking
Ainun Zuriyati Mohamed Asa'ari, J. Latifah, ... R.A. Ilyas
Pages 353-374
[Purchase](#) [View abstract](#) 

- Book chapter Abstract only
16 - Nanocellulose composites in the pulp and paper industry
Farah Nadia Mohammad Padzil, Ching Hao Lee, ... Hidayah Ariffin
Pages 375-395
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
17 - Nanocellulose nanocomposites in textiles
Mohd Azwan Jenol, M.N.F. Norrrahim and Norizan Mohd Nurazzi
Pages 397-408
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
18 - Nanocellulose as a bioadsorbent for water and wastewater purification
Mohd Idham Hakimi, Syed Umar Faruq Syed Najmuddin, ... R.A. Ilyas
Pages 409-437
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
19 - Nanocellulose composites in the automotive industry
Norizan Mohd Nurazzi, Mohd Azwan Jenol, ... A. Norli
Pages 439-467
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
20 - Advances in nanocellulose nanocomposites for bone repair
Innocent Jacob Macha
Pages 469-480
[Purchase](#) [View abstract](#) ▼

- Book chapter Abstract only
21 - Nanocellulose composites for electronic applications
A. Atiqah, F.A. Sabaruddin, ... M. Asrofi
Pages 481-502
[Purchase](#) [View abstract](#) ▼

- Book chapter Full text access
Index
Pages 503-524

Enhanced thermal stability of cellulose nanocrystals for processing polymer nanocomposites at a high temperature

13

Khairatun Najwa Mohd Amin

Faculty of Chemical and Process Engineering Technology, College of Engineering Technology, Universiti Malaysia Pahang, Lebuhraya Tun Razak, Kuantan, Pahang D.M., Malaysia

Chapter outline

13.1 Introduction 327

13.2 Process to enhance the thermal stability of CNC 328

13.3 Thermally stable CNC-reinforced polymer nanocomposites 330

References 333

13.1 Introduction

CNC has emerged as an attractive filler for polymer composites due to its intrinsic mechanical properties. Low loading of CNC between 0.5 and 1 wt% in the polymer matrix has demonstrated an extraordinary increase in tensile strength of various polymer composites for instance 20% and 35% of improvement in polyvinyl(acetate) (Nozaki & Lona, 2021) and polylactide (Chai et al., 2020) respectively. However, the process to fabricate CNC/polymer nanocomposites often involved conventional method which is not suitable for scalable production. A conventional method like solvent casting constitutes a problem in terms of production speed and environmental issues due to the high solvent usage (Jyoti, Basu, Singh, & Dhakate, 2015). Thus, the melt compounding method consists of extrusion and molding is preferred to suit industrial-scale production and demand. Melt compounding which is commonly employed by thermoplastic polymer operating at high processing temperature from as low as 120°C for common polyethylene (Zaaba & Ismail, 2019) up to 390°C for polymer-like Polyether ketone (Das et al., 2020). Table 13.1 offers an overview of techniques that were used recently to produce CNC-based composites with some respected examples.

One challenge in utilizing CNC in polymer composites specifically thermoplastic polymer fabricated through melt compounding method is that their thermal stability

Table 13.1 Techniques and fraction of CNC applied in CNC/polymer nanocomposites.

Technique	Polymer matrix	Fraction of CNC (wt%)	Ref.
Extrusion and/or molding	Polyethylene	1.5	Inai, Lewandowska, Ghita, and Eichhorn (2018)
	Polyethylene	1.0	Gray, Hamzeh, Kaboorani, and Abdulkhani (2018)
	Polypropylene	2.0	Sojoudiasli, Heuzey, and Carreau (2018)
	Polystyrene	10	Nagalakshmaiah, Nechyporchuk, El Kissi, and Dufresne (2017)
Solvent casting	Polyester	1–3	Zheng, Clemons, and Pilla (2019)
	Poly (lactic acid)	1.0	Hao et al. (2018)
	Waterborne Polyurethane	5	Mondragon et al. (2017)
	Poly (vinyl alcohol)	10	Popescu (2017)
	Acrylonitrile	1	Ma, Zhang, and Wang (2017)
	Butadiene Styrene		
	Rubber latex	1	Jailudin and Mohd Amin (2020)

which is close to melting temperature most of the polymer. This tends to rise degradation and discoloration of the extruded nanocomposites (Abhijit, Johannes, Sahlin-Sjöväld, Mikael, & Boldizar, 2020). Thus, recent researches are actively producing CNC with enhanced thermal stability to counter this problem.

13.2 Process to enhance the thermal stability of CNC

The most common isolation method to produce CNC is by using the sulphuric acid hydrolysis method. This method is preferable by most researchers due to its simple process flow, established protocols, and high production yield (2015). However, CNC produced has low thermal stability resulted by substitution of sulfate functional group over hydroxyl group which may be promoting dehydration reactions and act as flame retardants to the CNC. The onset degradation temperature mostly recorded values around 210–240°C (Zhang et al., 2020). Table 13.2 shows the onset degradation temperature of CNC according to the acid employed for the hydrolysis process.

Recently, ionic liquid (IL) also has been used to improve the thermal stability of CNC. IL is a green solvent with negligible vapor pressure which has multiple functions and initially is used in polymer modification. The attempt of ionic liquid-like 1-allyl-3-methylimidazolium chloride as a plasticizer has surprisingly improved the thermal stability of CNC (Liu, Guo, Nan, Duan, & Zhang, 2017). The results were obtained due to the ability of IL to partial desulfurization of CNC and the thermal degradation recorded at 240°C. Following that, a simple method of mixing IL with CNC was performed by rotary evaporate procedure, and it is able to increase the thermal

Table 13.2 Onset degradation temperature of CNC produced through acid hydrolysis.

Type of acid	Onset degradation temperature (°C)	Ref.
Sulphuric acid	200–240	Bano and Negi (2017), Xing, Zhang, Tu, and Hu (2018), and Zhang et al. (2020)
Hydrochloric acid	244	Hastuti, Kanomata, and Kitaoka (2018)
Phosphoric acid	260–310	(Espinosa, Kuhnt, Foster, & Weder, 2013), (Frost & Foster, 2020), Zhang et al. (2020), and (Wang et al., 2019)
Formic acid	300–325	Lv et al. (2019) and Du et al. (2017)
Mixed acids	250–300	Cheng et al. (2020), Vanderfleet et al. (2019), Wang, Yao, Zhou, and Zhang (2017), and Yu, Qin, Sun, Yan, and Yao (2014)

stability up to 210°C and increase the miscibility with polymer matrix as well (Song et al., 2018). Other than that, research also has shown that doing post alkali treatment on CNC film also has successfully erased the sulphate group resulting in a remarkable enhancement in thermal properties (Nan et al., 2017). This method successfully achieves thermal degradation at 260°C.

Alternatively, phosphoric acid or mixed acids (combination of two or more acids in hydrolysis process) have been utilized to isolate CNC with better thermal stability. Phosphoric acid, formic acid, and mixed acid are able to increase the thermal stability up to 300°C which is excellent to be reinforced with high-performance thermoplastic polymer with a processing temperature of more than 200°C (Das et al., 2020).

Other than that, mechanical methods like ultrasonication, homogenizer, and milling are also gaining high attention to be used in producing CNC. These methods are used as a posttreatment to produce CNC. CNC produce through this method provides good thermal stability as shown in Table 13.3 in fact it has a high production yield which is suitable for high scale production. In addition, this method also consumes low preparation time, acid/solvent usage, and energy (Mohd Amin, Annamalai, & Martin, 2017).

Overall, the degradation temperature of CNC able to be improved up to 300°C which falls in the temperature range of most high-performance thermoplastic polymer processing using the melt compounding method. This significant increase in thermal stability will boost up the employment of CNC as a reinforcing filler for polymer composite.

Table 13.3 Onset degradation temperature of CNC produced through a mechanical method.

Type of acid	Onset degradation temperature (°C)	Ref.
Ball milling	258–263	Mohd Amin, Annamalai, and Martin (2017)
Ultrasonication	250	Mohd Amin, Annamalai, Morrow, and Martin (2015)

13.3 Thermally stable CNC-reinforced polymer nanocomposites

Here the discussion on the CNC/polymer nanocomposite processed through melt compounding method is reviewed. The works are done to avoid thermal degradation of CNC either using preprocess to the CNC (grafting, modification, etc) or using ready thermally stable CNC to be reinforced with polymer and further process with melt compounding method with the purpose of high scalability production to suit the industry demand.

The first part will discuss the nanocomposites work which are using preprocess method to the low thermal stability of CNC. Polylactic acid (PLA) is one of the most promising biopolymers with exceptional properties, is being used in a wide variety of applications in diverse fields (Mao, Tang, Zhao, Zhou, & Wang, 2019). Research by Dhar, Tarafder, Kumar, and Katiyar (2016), has successfully fabricated CNC/PLA nanocomposite by using reactive extrusion. In this work, the CNC has been grafted with PLA using dicumyl peroxide as a crosslinking agent. Significantly, the grafted PLA chains shield the sulfate and hydroxyl groups of CNCs, thereby enhancing the compatibilization with the PLA matrix and preventing thermal degradation during extrusion. The extrusion processing temperature was at 180°C and the extrudate did not show any degradation or discoloration as shown in Fig. 13.1 with coding “PLAD CNC1”. With 1 wt% of CNC in the PLA matrix, the tensile strength of the CNC/PLA nanocomposite recorded 40% of improvement from neat PLA. A similar biopolymer which is PHBV (poly(3-hydroxybutyrate-co-3-hydroxyvalerate)), a family of bacterially-derived linear polyesters adopted a similar method (Zheng et al., 2019) as well. The maximum extrusion processing temperature is 160°C.

Next is polyamide which has garnered a lot of interest for its potential use in various applications ranging from deep-sea oil production to lightweight replacements for metals and rubbers in automobiles and aircrafts, to high-performance athletic shoes (Oliver-Ortega et al., 2019; Thokala, Kealey, Kennedy, Brady, & Farrell, 2017).

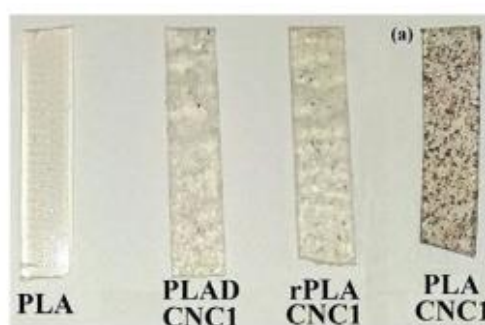


Fig. 13.1 Pictorial comparisons of the neat PLA, PLAD CNC1 and rPLA CNC1 strips with PLA/CNC 1 wt% extruded by simple melt blending.

Reproduction of image from Dhar, P., Tarafder, D., Kumar, A., & Katiyar, V. (2016).

Thermally recyclable polylactic acid/cellulose nanocrystal films through reactive extrusion process. *Polymer*, 87, 268–282. <https://doi.org/10.1016/j.polymer.2016.02.004> with permission from Elsevier.

Polyamide (PA) 12 has been reinforced with thermal stable CNC produced through phosphoric acid hydrolysis and was managed to be processed through melt compounding method (Niharat, Sapkota, Weder, & Foster, 2015). 1–20 wt% of CNC in the PA matrix was extruded at a temperature of 190°C and the tensile strength was increased from 40 to 72 MPa. Meanwhile a work with another type of PA which is PA 11 also able to process using melt compounding method as well by using preprocessing/premixing methods which is planetary ball milling and melt-compounding (rollerblade mixing) without the addition of processing aids or additional surface functionalization of CNC (Venkatraman, Gohn, Rhoades, & Foster, 2019). CNC used is from the acid hydrolysis process was further milled with PA 11. This process uses ball-bearing collisions to mechanically weld together two materials in the solid-state without solvents. Then, the PA12/CNC was melt compounded at temperature 190°C. The film fabricated as shown in Fig. 13.2, the method used able to prevent degradation of CNC as the film was compared with the sample without milling and just directly mixed and compound the PA11 and CNC. The tensile strength of the PA11/CNC with the milling process.

Another example of thermoplastic used with CNC is polypropylene (PP). PP can be fabricated to fiber and fabrics, film, injection, and blow molding products as well



Fig. 13.2 PA11 and PA11/CNC nanocomposite film fabricated via melt compounding with preprocess with milling (left) and without milling (right).

Reproduction of image from Venkatraman, P., Gohn, A. M., Rhoades, A. M., & Foster, E. J. (2019). Developing high performance PA 11/cellulose nanocomposites for industrial-scale melt processing. *Composites Part B: Engineering*, 174, 106988. <https://doi.org/10.1016/j.compositesb.2019.106988> with permission from Elsevier.

Index

Note: Page numbers followed by *f* indicate figures and *t* indicate tables.

A

- Acetate butyrate, 380
- Acetylacetone sensor, 220
- Acetylation, of CNF, 296
- Acid hydrolysis, 3, 9–10, 65–66, 440–441, 485–486
- Acids, 412, 414*t*
- Acoustic wave-based sensor, 215
- Activated carbon, 415–416
- Active cooling system, 482
- Active packaging, 320–323
- Acute toxicity
 - dermal toxicity safety study, 252–254
 - inhalation toxicity safety study, 252–253
 - oral toxicity safety study, 252
- Additives, 376
- Adhesives, nanocellulose
 - bio-based resins (*see* Bioresins)
 - from plant biomass sources, 69
 - properties, 69
 - solid content and viscosity, increase in, 69
 - thermoplastic resins
 - hot melts, 75–76, 76*f*
 - polyvinyl acetate (PVA) adhesives, 76–77, 77*f*
 - thermosetting resins
 - aminoplastic adhesives, 69
 - condensed adhesives, 69
 - formaldehyde, 69–70
 - isocyanate adhesives, 74–75, 75*f*
 - melamine formaldehyde (MF) adhesive, 73–74, 74*f*
 - melamine urea formaldehyde (MUF) adhesive, 73
 - phenolic resins (*see* Phenol-formaldehyde (PF) resin)
 - phenol resorcinol formaldehyde (PRF) adhesive, 74
 - resorcinol formaldehyde (RF) adhesive, 74, 74*f*
 - urea formaldehyde (UF) adhesives (*see* Urea formaldehyde (UF) adhesives)
- Adsorbents
 - activated carbon and zeolites, 198
 - nanocellulose, 199–204, 202*t*, 205–207*t*
- Adsorption
 - definition, 198
 - dye, adsorption kinetics of, 401–402
 - heavy metals (*see* Heavy metals, as chemical contaminants)
- Aerogels
 - advantage of, 139–140, 201
 - applications, 139–140
 - definition, 139–140, 201
 - polymer aerogels, 148
 - applications, 141, 142*f*
 - biopolymers/natural polymers, 140, 143
 - carbon/carbide-based aerogels, 141
 - cellulose and nanocellulose-based aerogels (*see* Cellulose, aerogels)
 - cross-linked chemical structure, 140, 141*f*
 - definition, 140
 - hybrid aerogels, 141
 - inorganic/organic aerogels, 141
 - macro and microscale, microporous structure, 140, 141*f*
 - synthesis, stages for, 141–142, 142*f*
 - synthetic polymers, 140–143
 - properties, 139–140
- Ag/HMT composites, 182–187, 185*f*, 186*t*
- Agriculture, nanocellulose hydrogels in, 274–275
- Alcohol fuel cell sensor, 218
- Aldehyde gases, 217
- Algae, 313
- Alginate, 340–341
- Alginate-CNC hydrogels, 269, 273
- Alginate-MoO₃ NPs, 219
- Alginate-NCC composite, 363–364
- Alkali treatment, 10, 11*f*, 13
- Alpha-tocopherol, 321–323, 321*t*
- Aluminum metal matrix composites, 457–458

- Ames test, 170
Aminoplastic adhesives, 69, 78–79
Amino silane-modified nanocellulose, 182, 183*t*, 184*f*
Ammonia-based pretreatment, 10
Ammonia gas sensors, 217–218
Ammonia sensor, 215
Ammonium persulfate (APS) oxidative agent, 163–164
Anionic nanofibrillar cellulose (aNFC) hydrogel, 342
Antimicrobials materials, 400
Antioxidants, in packaging materials
 natural antioxidants, 320–323
 synthetic antioxidants, 320–321
Asbestos fibers, 248
Ash, 57
Atomic force microscopy (AFM), 19–20
AuNP/BNC nanocomposites, 229, 233–234
Autologous, for bone loss, 469–470
Automotive applications, nanocellulose, 460
 advantages, 443
 beneficial characteristics, 440–442, 441*f*
 bionanocomposites, 21–23, 25, 25*f*
 bumper, structuring of, 442
 crystal modulus of, 443
 desulfation method, 442–443
 electric vehicles, 456
 features, 443
 green materials, future projection of, 454–455
 Kyoto technique, 456
 lightweight materials, future projection of, 454–456, 454*f*
 lignocellulose residues, 456–457
 limitations, 442
 metal-matrix composites, 457–460
 nanocellulose nanocomposites (*see* Nanocellulose nanocomposites (NNC))
 plant fibers, 454–455
 sheet molding compound (SMC) composite, 441–442
 surface chemical modification, 442–443
 surface functionalization, 443
 thermosetting polymers
 as composite matrix, 451–452
 nanocellulose reinforced epoxy composite, 452–453
 natural fiber reinforced thermoset composites, 453
 vehicle components, usage in, 441–442
- B**
Bacterial cellulose nanocrystal (BCNC), 219
Bacterial cellulose (BC) nanoribbons. *See* Bacterial nanocellulose (BNC)
Bacterial nanocellulose (BNC), 2–3, 3*f*, 52, 59*f*, 160, 214–215, 264–265, 316–317, 354, 376, 380, 382, 398, 440, 472–473
 aerogels, 140, 144
 bacteria, 486
 biomedical applications, 26, 68, 337–338, 487
 biocompatibility and stability, 339, 470–471
 bone repair and regeneration, tissue engineering for, 471–472
 cytocompatibility, 339
 wound dressing applications, 343, 402–403
 characteristic of, 4–5, 5*t*
 commercialization of, 487
 copper oxide (CuO) nanoparticles, 99–100
 definition, 4
 degree of polymerization (DP), 6–7
 drawbacks, 486–487
 electron microscope images of, 317, 318*f*
 enzymatic hydrolysis, 483
 extraction method, 483, 484*t*
 extraction sources, 8*t*
 food application, 164–165, 487
 gas sensors, 218–219
 glucose biosensor, 234
 hydrogen peroxide (H₂O₂) biosensor, 233–234
 isolation of, 164
 lyophilized fabric-reinforced CS/BNC, bacteriostatic effect, 400–401
 mechanical properties, 7
 morphology of, 6
 preparation methods, 361
 production, 469–470
 from bacteria species, 4, 67
 continuous cultivation methods, 68

- static cultivation method, 68
- sugar-rich novel substrates, 67–68
- properties, 67, 483, 484*t*, 486–487
- strain sensors, 226
- synonyms, 58–59
- toxicology properties, 470–471
- Ball milling, 329–331, 329*t*
- Banana pseudostem fibers, 384
- Bearberry extract, 322–323
- Benzaldehyde (BZH) chemical sensor, 220
- Beta-cyclodextrin (beta-CD), 221
- Bimetallic nanoparticles (BNPs), 220–221
- Bioadsorbent materials, 410
 - activated carbon, 415–416
 - chitosan, 416–417
 - clay minerals, 416
 - nanocellulose
 - as bioadsorbent, 410, 420–430
 - functionalization, 417–418
 - nanomaterials, 410, 417
- Biocomposite packaging film, 321–323
- Biocomposites
 - in automotive application, 443–444
 - critical issues, 445
 - fuel consumption and CO₂ emissions, reduction of, 444
 - lightweight biocomposites, use of, 444
 - natural fibers, polymeric materials reinforced with, 445
 - in food packaging, 357–364
 - high durability paper, 364–367
 - non-structural biocomposites, 356–357
 - printing paper, 367–369
 - structural biocomposites, 356–357
 - wood-based composites (*see* Wood-based composites)
- Biodegradable films, 311–312
 - advantages, 312
 - biodegradable guar gum/nano-crystalline films, 318
 - biopolymers, 312
 - cellulose (*see* Cellulose)
 - definition, 312
 - as packaging materials, 312
 - preparation, 312
- Biodegradable polymers
 - classification of, 358, 359*f*
 - in food packing, 357–359, 360*t*
- Bioink, 340–341
- Biological wastes, nanocellulose from, 14, 15*f*, 16*t*
 - ginger, 15, 17*f*
 - kenaf, 16–18, 18*f*
 - sugarcane bagasse nanocellulose (SBCN), 19, 19*f*
 - sugar palm fiber (SPF), 10, 11*f*, 19–20, 20*f*
 - water hyacinth, 16, 18*f*
- Biomedical applications, nanocellulose in, 52, 160, 347
 - bacterial nanocellulose (BNC) (*see* Bacterial nanocellulose (BNC))
 - biological implants, 339–341
 - biomedical materials, biological properties of, 338–339
 - bionanocomposites, 26, 26*f*
 - cellulose nanocrystals (CNC) (*see* Cellulose nanocrystals (CNC))
 - cellulose nanofibers (CNF) (*see* Cellulose nanofibers (CNF))
 - drug delivery (*see* Drug delivery system)
 - tissue engineering (*see* Tissue engineering (TE))
 - wound dressing (*see* Wound dressing applications)
- Bionanocomposites, 21–27, 22*t*
 - automotive applications, 21–23, 25, 25*f*
 - biomedical applications, 26, 26*f*
 - biopolymers (*see* Biopolymers)
 - flexible optoelectronic, 25–26, 25*f*
 - food packaging application, 357–364
 - food packaging applications, 21, 23–24*f*
- Bionanomaterials
 - hemicelluloses, 61–63
 - lignin, 60–61
 - nanocellulose (*see* Nanocellulose (NC))
- Biopolymers
 - aerogels, 140–143
 - bionanocomposites fabrication, 356
 - cellulose (*see* Cellulose)
 - definition, 358
 - in food packing
 - biodegradable polymers, 357–359, 360*t*
 - lignocellulosic materials, 359
 - nanocellulose-based composites, 361–364, 362*t*
 - hydrogels, 268–269
 - packaging materials, 312

Biopolymers (Continued)

- carrageenan, 313–315
- cellulose (*see* Cellulose)
- plasticizers, 319–320
- starch, 312

Bioresins, 77

- animal waste, adhesives from, 81
- blood albumin adhesives, 80
- casein adhesives, 80–81
- lignin, 77–78, 78*f*
- soy protein adhesive, 79–80
- tannin-based adhesives, 78–79, 79*f*

Biosensors

- components of, 226–227, 227*f*
- definition, 226
- nanocellulose application in
 - electrical biosensors, 233–234
 - optical biosensors (*see* Optical biosensors)

Blood albumin adhesives, 80**Bone substitute materials, 469–470****Bone tissue regeneration and repair**

- autologous, for bone loss, 469–470
- bone substitute materials, development of, 469–470

cellulose nanocomposites

- cellulose nanocrystals-hydrogel nanocomposites, 474–476
- CNC nanocomposites, 472–473, 473*f*
- CNC with maleic anhydride grafted polylactic acid, 474–476
- fabrication techniques, 473–474, 475*f*
- hydroxyapatite-bacterial cellulose (HAP-BC) nanocomposite, 474–476
- otolith/collagen-BC nanocomposites, 474–476
- for tissue engineering applications, 474–476, 477*t*

natural polymer nanocomposites, 469–470**tissue engineering (TE), 469–472****Bone tissue scaffolding, CNC aerogels for, 145–146****Boron nitride (BN) nanosheets, 399–400, 399*r*****Breathalyzers, 219****Brunauer-Emmett-Teller (BET), 19–20****Butylated hydroxyanisole (BHA), 320–321****Butylated hydroxytoluene (BHT), 320–321****C****Capital expenditure (CAPEX), 41, 45, 46*r*****Carbide-based aerogels, 141****Carbon black (CB), 25, 220****Carbon dioxide (CO₂) emission, 442, 444, 447–448****Carbon nano-dots (CNDs), 219–220****Carbon nanofibrils (CNFs), 427, 429****Carbon nanostructures (CNSs), 219–220****Carbon nanotubes (CNTs), 105, 220, 225, 417****Carbon-neutral lignocellulosic materials, 50****Carbon quantum dot/nanofibrillated cellulose (CQDs/NFC) composite aerogel, 202–203, 203*f*****Carbon quantum dots (CQD), 222****Carboxylation, 91–95****Carboxymethylated chitosan, 427****Carboxymethyl cellulose (CMC), 165****Carboxymethyl cellulose hydrogels, 269****Carrageenan****iota-carrageenan**

- 3,6-anhydrogalactose content, 313, 314*f*
- ester sulfate content of, 313, 314*f*
- food applications, 313
- gelation properties, 313
- structure, 313, 314*f*

kappa-carrageenan

- 3,6-anhydrogalactose content, 313, 314*f*
- ester sulfate content of, 313, 314*f*
- food industries, used in, 313
- gelation properties, 313
- structure, 313, 314*f*

lambda-carrageenan

- 3,6-anhydrogalactose content, 313, 314*f*
- ester sulfate content of, 313, 314*f*
- food applications, 313
- gelation properties, 313
- structure, 313, 314*f*

seaweeds, 313**semirefined carrageenan film, 323****Casein adhesives, 80–81****Casting methods**

- aqueous casting solution, 378–379
- nonaqueous casting solution, 378–380

CellF-carbon nanotube (CNT) aerogel, 225**Cellobiases, 10–12****Cellulase, types of, 10–12****Cellulose, 2–3, 50–51, 482**

- aerogels, 144–145, 148
 - advantages, 140, 143–144, 201
 - bacterial nanocellulose (BNC), 140, 144
 - cellulose nanocrystals (CNC), 140, 145–147, 145*f*
 - cellulose nanofibers (CNF), 140, 145, 145*f*, 147–148
 - characteristics, 140, 143–144
 - hydroxyl groups, presence of, 143–144
 - regenerated cellulose, 140, 144
 - sources, 144
 - synthesis, steps for, 144
- annual worldwide production, 51
- applications, 214
- biodegradable films, raw material for, 314–315
- biodegradation, 470–471
- biomedical application, 337–338
- biomedical materials, biological properties of, 338–339
- chain, molecular structure of, 54, 55*f*
- characteristics, 155–156, 264, 397
- crystalline cellulose, 157–158
- degree of polymerization (DP), 155–156
- fibers, 445
- food packaging applications, 359
- gas sensors, 216
- glucose units, 51, 155–156
- hydrogels, 264, 342
- insolubility of, 315
- intramolecular and intermolecular hydrogen bonding networks, 314–315, 315*f*
- mechanical properties, 51
- molecular weight of, 55
- nanomaterials (*see* Nanocellulose (NC))
- natural cellulose, 155–156
- from natural resources, 469–470
- OH groups, 54–55
- oil palm biomass (OPB) (*see* Oil palm biomass (OPB))
- oxidized cellulose, 470–471
- from plant fibers, 2
- properties, 51, 214, 314–315
- and seaweed, compatibility between, 318–319
- sources, 58, 315, 316*t*
- stereochemical formula of, 54, 55*f*
- as structural polymer, 155–156
- structure, 156–157, 157*f*, 439–440
- yield, 54
- Cellulose-based aerogels (CAGs), 429–430
- Cellulose nanocomposites, 487–488
 - bone repair and regeneration (*see* Bone tissue regeneration and repair)
 - definition, 472–473
- Cellulose nanocrystals (CNC), 2–3, 3*f*, 52, 59*f*, 160, 214–215, 234–235, 264–265, 317, 354, 376, 398, 440, 472–473
 - acid hydrolysis, 65–66
 - adsorption, 203–204
 - aerogels, 140, 145–147, 145*f*
 - applications, 5, 67, 486
 - in automotive applications, 441–442, 460
 - biocompatibility properties, 470–471
 - biomedical applications, 26, 337–338
 - bone repair and regeneration, tissue engineering for, 471–476, 473*f*
 - drug delivery, 341
 - in vitro cytotoxicity studies, 339
 - tri-dimension (3D) of electrospun scaffolds, 342
- biopolymer composites, for packaging applications, 361, 362*t*
 - alginate, 363–364
 - chitosan, 363
 - glycerol-plasticized wheat starch, 361–363
 - polyhydroxyalkanoates (PHA), 364
 - polyhydroxybutyrate (PHB), 364
 - polylactic acid (PLA), 363
 - whey protein isolate, 364
- carbon nanoparticles, 106
- characteristic of, 4–5, 5*t*
- chemical composition, 3
- chemical hydrolysis, 440
- cytotoxicity studies, 249–250, 251*t*
- definition, 3
- electron microscope images of, 317, 318*f*
- extraction method, 3, 58–59, 356, 483, 484*t*, 485–486
- extraction of, 316–317
- extraction sources, 8*t*
- food application, 164–165
- functionalized ZnO/CNC, bacteriostatic effect of, 400–401
- gas sensors

- Cellulose nanocrystals (CNC) (*Continued*)
- ammonia gas sensors, PANI/CNC composites, 217
 - CNC-modified tin dioxide films, 217
 - colorimetric sensor, 217
 - electrode sensor, 217–218
 - nitrogen dioxide (NO₂) gas sensor, 216–217
 - hydrogels, 265–268
 - alginate hydrogels, 269, 273
 - carboxymethylcellulose/hydroxyethylcellulose hydrogels, 269
 - chitosan hydrogels, 269
 - gelatin hydrogels, 269
 - polyacrylamide (PAAM) hydrogels, 268
 - polyacrylic acid (PAA) hydrogels, 268
 - poly (ethylene glycol) (PEG) hydrogels, 268
 - poly (vinyl alcohol) (PVA) hydrogels, 268
 - sodium alginate/gelatin hydrogels, 269
 - tissue engineering applications, 271
 - wound dressing application, 271–272
 - isolation of, 161–164, 440–441
 - kenaf, 16–18, 18*f*
 - mechanical properties, 7, 215
 - as mechanical reinforcing agent, 486
 - morphology of, 6, 361
 - paper coating process, 380
 - particle behavior of, 67
 - polymer composites/nanocomposites
 - challenge, 327–328
 - solvent casting method, 327, 328*t*, 332, 332*f*
 - thermoplastic polymer, melt compounding, 327, 328*t*, 329–333
 - preparation, 5, 361
 - production, 469–470
 - production methods, 377
 - properties, 65–66, 483, 484*t*, 485–486
 - “rice-like shape”, 4*f*
 - short-rod-like/whisker shape, 3
 - size of, 3, 65, 440, 485
 - strain sensor
 - CNC-grafted GO hybrid materials, 225
 - CNTs/CNC sensitivity, 226
 - surface modification, 67
 - synonyms, 3, 58–59, 65, 483, 484*t*, 485
 - thermal stability, enhancement of, 7, 329
 - CNC film, post alkali treatment on, 328–329
 - ionic liquid (IL), 328–329
 - mechanical methods, 329, 329*t*
 - phosphoric acid, formic acid, and mixed acid, 329
 - sulphuric acid hydrolysis method, 328, 329*t*
 - toxicological report for, 470–471
 - urea-formaldehyde adhesive, interaction with, 87–89
 - yield, 66–67
- Cellulose nanofiber boards (CNFB), 252–253
- Cellulose nanofiber in water (CNFW)
- chemical identity and nano-properties of, 246, 248*t*
 - eye irritation study, 253
 - oral toxicity study, 252
 - skin corrosion/irritation and skin sensitization, 253–254
- Cellulose nanofibers (CNF), 2–4, 3*f*, 52, 59*f*, 87–89, 160, 214–215, 264–265, 316–317, 354, 376, 378, 398, 440–442, 469–470, 472–473
- aerogels, 140, 145, 145*f*, 147–148
 - applications, 289–290, 485
 - in automotive applications, 441–442, 460
 - biological safety studies, 245–246, 257
 - aquatic test system, 254–256
 - cellular test system, 249–250
 - industry and consumer products, application of, 247–249
 - mammalian test system, 250–254
 - physicochemical characterization, CNF suspension, 246
 - biomedical applications, 337–338
 - bone repair and regeneration, tissue engineering for, 471–472
 - drug delivery, 341
 - 3D printing bioink, 340–341
 - wound dressing applications, 343–344
 - biopolymer composites, for packaging applications, 361, 362*t*, 363
 - characteristic of, 4–5, 5*t*
 - chemical modification of, 65
 - chemical pretreatment, 64–65
 - definition, 483–485
 - development of, 289–290
 - drawbacks, 63–64

- electrical biosensors, 233
- electron microscope images of, 317, 318*f*
- enzymatic pretreatment, 12, 64–65
- extraction methods, 63–64, 483, 484*t*, 485
- extraction processes, 317
- extraction sources, 8*t*
- fabrication, 356
- food application, 164–165
- gas sensors, 218
- high energy consumption and production expense, 485
- high-pressure homogenization, 356
- hybrid CQD/CNF, 222
- hydrogels, 266–268
 - alginate hydrogels, 269
 - anionic nanofibrillar cellulose (aNFC) hydrogel, 342
 - chitosan hydrogels, 269
 - collagen hydrogels, 269
 - poly (ethylene glycol) (PEG) hydrogels, 268
 - poly (vinyl alcohol) (PVA) hydrogels, 268
 - wound dressing application, 271
- hydroxyapatite (HAP) composite foam, 101
- isolation of, 161
- mechanical methods, 289–290
- mechanical properties, 7
- microfluidization, 356
- morphology of, 6, 361
- “noodle-like shape”, 4*f*
- paper coating process, 380
- papermaking applications, 356
- particleboard composites, performance enhancement of, 87–89
- polymer composites, reinforcement material for (*see* Polymer reinforced CNF composites)
- preparation methods, 361
- printing paper
 - impacts and advantages, 368–369
 - paper strength, 368
- production, from OPMF, 47
 - characterization analysis, 41
 - economic analysis, 41
 - economic evaluation, by SHS-WDM and KOH-WDM, 44–45
 - hemicellulose removal, SHS and KOH pretreatments for, 40–41
 - mass balance, by SHS-WDM and KOH-WDM, 43, 43*f*
 - materials, 40
 - mechanical fibrillation, 41
 - SHS- and KOH-treated CNFs, properties of, 41–43, 42*t*, 43*f*
 - untreated, SHS- and KOH-treated pulps, properties of, 41–42, 42*t*
 - production methods, 376–378
 - properties, 289–290, 483, 484*t*
 - silylated NFC reinforced PP composite, 490
 - size of, 4, 63, 483–485
 - strain sensor
 - GN-CNF@PVA hydrogel, 225
 - polyvinyl alcohol (PVA)/CNF hydrogel, for pressure/strain sensing, 224
 - sugar palm fiber (SPF), 20
 - synonyms, 4, 58–59, 63–64, 483–485, 484*t*
 - thermal conductivity (TC) properties, 399*t*
 - Ag-rGO/NFC, 399–400
 - CNF/OH-BNNS, 399
 - 2D-layered NFC/BN, 399–400
 - thermal stability, 7
 - thin film transistors (TFTs), 494–496
 - toxicological report for, 470–471
 - urea-formaldehyde adhesive, particleboard panels bonded with, 87
- Cellulose nanowhiskers (CNWs). *See* Cellulose nanocrystals (CNC)
- Centrifugal casting molding method, 106
- CeO₂ nanoparticles, 220
- Ceramic materials, 458
- Cetyl triammonium bromide (CTAB), 106
- Cetyltrimethylammonium bromide (CTMAB), 490
- Chemical contaminants
 - acids, 412, 414*t*
 - dyes, 411, 426–428
 - heavy metals, 411, 420–426
 - organic oils, 412, 428–430
 - organochlorines, 412, 414*t*
 - organophosphorus, 412, 414*t*
 - phenols, 412, 414*t*
 - plant nutrients, 412, 414*t*
 - point and nonpoint sources, 411
- Chemically engineered nanocellulose (CMNC), 222
- Chemical modification, 168–169

- Chemical pretreatment methods, 9–10, 12
- Chemical sensors
- definition, 219
 - metal oxide, bimetal, bimetal oxide
 - chemical sensor, 220–222
 - nanocellulose chemical sensor, 222–223
 - transduction methods, 219
 - type of, 219–220
- Chemical vapor deposition (CVD) method, 429–430
- Chemisorption, 415–417
- Chemithermomechanical (CTMP) paper sheets, 382
- Chitosan (CS), 400–401, 416–417
- Chitosan aerogel, 202–203
- Chitosan hydrogels, 269
- Chitosan-NCC composite, 363
- Chitosan/poly(vinylpyrrolidone)/nanocellulose (CPN), 403
- Chlorhexidine digluconate, 272
- Chloropropyltriethoxysilane (CPTES), 146–147
- Cholesterol sensor, 222
- Chromium (Cr), 202–203
- Citric acid-modified cellulose nanofibrils, 421–425
- Clay minerals, 416
- Clofazimine, 272
- Coating materials, nanocellulose
- nanocomposites in, 192
 - air resistance, 187–189, 189f
 - applications, 192, 193f
 - corrosion resistance, 189–190
 - crack resistance, 190, 191f
 - hemp fiber, 180
 - mechanical resistance properties
 - abrasion resistance, 187
 - HMT nanocomposites, coatings
 - modified with, 185–187, 186f
 - impact strength, 187
 - tensile strength, 187
 - modified nanocellulose coating
 - on glass and aluminum substrates, 182
 - on steel substrates, 182–184
 - polarized optical microscopy, 190–191, 191f
 - silane, surface modification with, 180–182, 181f
 - water absorption properties, 189, 190f
 - water uptake and permeability, 191–192, 192f
- Coefficient of OLEDs' thermal expansion (CTE), 496
- Collagen-nanocellulose hydrogels, 269
- Colorimetric biosensors, nanocellulose-based, 231–233
- Colorimetric sensor, 217
- Compatibilizer, 296–298, 297f
- Composites, nanocellulose reinforcement in
- advantages, 81–82
 - polymer composites/nanocomposites, 107–109
 - applications, 89
 - carbon-derived nanoparticles, 90
 - conductive nanomaterials, 90
 - mechanical properties of, 487–490
 - metal-based nanocomposites, 90–95, 92–94f
 - metal oxide/inorganic based nanocomposites, 95–101, 96–98f
 - nanocarbon based nanocomposites, 101–107, 102–104f
 - problems of, 108
 - production techniques, 108
 - significance, 81–82
 - wood-based composites (*see* Wood-based composites)
- Compressible and elastic carbon aerogels (CECAs), 226
- Compression molding, 302
- Conductive nanocellulose composites, 491–492
- Conductive nanomaterials, 90
- Conductive nanoparticles, 223
- Conductive polyaniline/CNC (PANI/CNC) composites, 217
- Conductive polymers, 223–224
- Confocal laser filtering magnifying instrument (CLSM), 165–166
- Continuous cultivation methods, 68
- Copper oxide nanoparticles (CuO NPs), 99–100
- Corrosive hydrolysis, 161–162
- Cosmetics
- definition, 275
 - nanocellulose hydrogel, 275–276
- Cotton cellulose nanocrystals, 230–231, 231f
- C-periodate nanocellulose, 403

- Crude oil sensing, 219–220
Crude palm oil production, 39–40
Crystalline cellulose, 157–158
Crystallinity index, 41–42
Cytotoxicity studies, of nanocellulose, 249–250, 251*t*
- D**
- Daphnia magna* reproduction test, 254–255, 256*f*
Deep eutectic solvents (DESs), 12
Degree of polymerization (DP), 6–7, 57, 155–156
Desulfation method, 442–443
Dietary fiber, nanocellulose as, 164–168
Dilute-acid hydrolysis, 9–10
Dimethylol phenol, 70
Dip-coating procedure, 226
Discontinuously reinforced aluminum (DRA) composite, 458
Discontinuously reinforced metal-matrix composite, 458
Dissolved gas analysis (DGA), 215–216
Dopamine (DA), 341
Double pickering emulsions, 166
Double-walled carbon nanotubes (DWCNTs), 226
Drop-casting method, 232–233
Drug delivery system, 341
 definition, 272
 nanocellulose hydrogel, 272–273
Dye contaminants
 adsorption kinetics, 401–402
 nanocellulose treatment, 426–428
 types, 411, 413*f*
Dynamic mechanical analysis (DMA), 295
- E**
- Ecotoxicity study, 254–256
Electrical biosensors, nanocellulose-based, 233–234
Electric vehicles, 456
Electrochemical sensors, 215, 220
Electro-conductive hydrogels (ECHs), 223–224
Electrode sensor, 217–218
Electro impedance spectroscopy (EIS), 189–190
Electronic applications, nanocellulose in challenges, 497
nanocellulose composites
 conductive composites, 492, 493*t*
 electrical properties of, 491
 lithium-ion batteries, separator of, 496–497
 mechanical properties, 487–490
 organic field-effect transistors (OFETs), 496
 organic light-emitting diodes (OLEDs), 496
 piezoelectric and electroactive effect, 492–494
 supercapacitors (SC), 494, 495*t*
 thermal properties, 490–491
 thin film transistors (TFTs), 494–496
 properties of, 497
 temperature and pressure sensor, 492
Electronic devices
 electronic material devices, thermal management of, 482
 heat dissipation mechanism, 482
 microprocessors for, 482
Electrospinning technique, 161, 342, 474
Electrostatically settled nanocrystalline cellulose (ENCC), 169–170
Emulsions, 165–166
Endocellulases, 10–12
Endothelial cells (ECs), 340
Energy storage systems, 459–460
Enzymatic hydrolysis, 161–162, 483
Enzymatic pretreatment, 10–12
Epichlorohydrin (ECH), 144
Epoxy, 451–452, 452*f*
Epoxypropyltrimethylammonium chloride, 443
Epoxy silane, 182, 184*f*
Esterification, of CNF, 296
Ethylene-vinyl acetate (EVA) copolymer, 75–76, 76*f*
Ethylene vinyl alcohol (EVOH), 168
Exocellulase, 10–12
Extractive substances, 50, 57–58
Extrusion process, 298–300, 299*f*
Eye damage/irritation safety study, 253
- F**
- Ferroferric oxide (Fe₃O₄) nanoparticles, 202–203
Fiber-based wood composites, 82, 83*f*, 85

- Field emission scanning electron microscopy (FESEM), 19–20
- Fillers, 376, 378
- Fish acute toxicity test, 255–256
- Fish embryo acute toxicity (FET) test, 255–256
- Fluorescent biosensor, nanocellulose-based, 230–231
- Food industry, nanocellulose in, 171
 - dietary fiber, 164–168
 - emulsifying and stabilizing agent, 165–166
 - food additive, 164–165
 - food packaging (*see* Packaging, nanocellulose in)
 - hydrogels, 273–274
 - safety, 170
- Food Standards Agency (FSA), 248–249
- Formaldehyde, 69–70
- Formic acid, 329
- Fourier transform infrared spectroscopy (FT-IR), 19–20
- Fracture morphology analysis, 25
- Freeze-drying process, 141–142, 474
- Functional Food Center (FFC), 167–168
- Functional food ingredient, 166–168
- Functional Food Science in Europe (FuFoSE), 167
- Functionalized nanocellulose, 417–418, 419*f*
- ## G
- Gas sensors, 215–216
 - bacterial nanocellulose (BC), 218–219
 - based on nanomaterials, 216
 - cellulose, 216
 - cellulose nanocrystals (CNC)
 - ammonia gas sensors, PANI/CNC composites, 217
 - CNC-modified tin dioxide films, 217
 - colorimetric sensor, 217
 - electrode sensor, 217–218
 - nitrogen dioxide (NO₂) gas sensor, 216–217
 - cellulose nanofibers (CNFs), 218
 - parameters, 216
 - portable gas sensor, function of, 216
 - principle of, 216
- Gas-stage hydrochloric corrosive hydrolysis, 161–162
- Gelatin/BHA mats, 320–321
- Gelatin film packaging, 322–323
- Gelatin-nanocellulose hydrogels, 269
- Generally regarded as safe (GRAS), 170
- Genotoxicity, of CNF, 249–250
- Ginger nanocellulose, 15, 17*f*
- Glass fiber (GF)/epoxy composite, 441–442
- Glassy carbon electrode (GCE), 220, 233–234
- Global Harmonized System (GHS), 246, 250–253
- Global warming, 446
- Glucose, 50–51
- Glucose biosensor, 234
- Glucose oxidase (GOx), 234
- Glue-laminated timber (Glulam), 86–89
- Glycerol, as plasticizer, 320, 320*t*
- GN-CNF@PVA hydrogel, 225
- Graft-co-polymerization technique, 222
- Graphene (GN), 225
- Graphene oxide-silica (GO-silica), 427
- Graph oxide (GO), 218
- Greenhouse gas emission, 446–447
- Green tea extract, 323
- ## H
- Heavy metals, as chemical contaminants, 197, 199–200
 - adsorption method, 198, 200
 - health effects, 411, 412*t*
 - nanocellulose as adsorbent, 199, 205–207*t*, 420–426
 - challenges, 204
 - chitosan aerogel, 202–203
 - CQDs/NFC composite aerogel, 202–203, 203*f*
 - ferroferric oxide (Fe₃O₄) nanoparticles, 202–203
 - functionalization, 199, 204
 - hydrogel composites, 203–204
 - nanopaper ion-exchanger, 204
 - properties, 200–201, 202*t*, 204
 - response surface methodology, 202–203
 - point/nonpoint sources, 199
 - remediation
 - conventional approaches, 198
 - lignocellulosic biomass for, 198–199
 - water sources, 199–200
- Hemicellulose, 50–51, 56, 439–440
 - applications, 62–63
 - classification, 56–57

- extraction, 62
 - molecules vs. cellulose molecules, 57
 - nanohemicellulose, 63
 - properties, 62
 - in wood, 55, 61–62
 - Hemp fiber, 180
 - Hexamethylenetetramine, 78
 - High-density polyethylene (HDPE), 290–291, 291*t*
 - High durability paper
 - nanocellulose application
 - advantages, 365
 - challenges, 366
 - incorporation of, 366–367
 - promising results in production, 367
 - for specialty applications, 364–365
 - synthetic polymers, 365
 - High-pressure homogenization (HPH), 13–14, 20
 - Hornification, 201
 - Horseradish peroxidase (HRP), 233–234
 - Hot melt adhesives
 - applications, 75
 - characteristics, 75
 - ethylene-vinyl acetate (EVA) copolymer, 75–76, 76*f*
 - features, 75
 - high viscosity, 75–76
 - polyamide adhesives, 76
 - Hydrochloric acid hydrolysis, 66
 - Hydrocolloid gums, 78–79
 - Hydrogels
 - cellulose, 264, 342
 - characteristics, 263
 - composites, 203–204
 - crosslinked polymer, 263–264
 - definition, 263
 - GN-CNF@PVA hydrogel, 225
 - interpenetrating polymer networks (IPNs), 263–264
 - nanocellulose, 264–265, 276–277
 - in agriculture, 274–275
 - challenges, 277
 - CNC hydrogel, 265–266
 - in cosmetics, 275–276
 - drug delivery system, 272–273
 - food applications, 273–274
 - NFC hydrogels, 266–267
 - reinforced hydrogels, 267–270
 - tissue engineering applications, 270–271, 342
 - wound dressing application, 271–272
 - natural polymers, 263–264
 - polyvinyl alcohol (PVA)/CNF hydrogel, 224
 - preparation, 263
 - self-healing hydrogels, 224
 - synthetic polymers, 263–264
 - Hydrogenated acrylonitrile-butadiene rubber (H-NBR) composites, 490–491
 - Hydrogen peroxide (H₂O₂) biosensor, 233–234
 - Hydrogen sulfide (H₂S) gas sensor, 216, 219
 - Hydrolysis, 161–164, 180–181
 - Hydrophobic nanoclay (HMT)
 - nanocomposites, 182–187, 185*f*, 186*t*
 - Hydrophobic polymers, 448
 - Hydroxyapatite-bacterial cellulose (HAp-BC) nanocomposite, 474–476
 - Hydroxyapatite (HAp) nanoparticles, 218
 - Hydroxyethylcellulose hydrogels, 269
 - Hydroxylated boron nitride nanosheets (OH-BNNS), 399, 399*t*
 - Hypoxemia, 248–249
- ## I
- Inhalation toxicity safety study, 252–253
 - Injection molding process, 25, 301–302
 - Inorganic aerogels, 141
 - Inorganic substances, 57
 - In-situ chemical deposition process, 95
 - Institute of Food Technologists (IFT), 167
 - Institutional Animal Care and Use Committee (IACUC), 252
 - Interlaminar shear strength (ILSS), 451–452, 452*f*
 - Internal bond strength (IB), 87–89
 - Internal melt-blending, 300–301, 300*f*
 - Interpenetrating polymer networks (IPNs), 263–264
 - Ionic liquid-like 1-allyl-3-methylimidazolium chloride, 328–329
 - Ionic liquids (IL), 12
 - Iron gall ink, 382
 - Iron oxide/HMT composites, 182–187, 185*f*, 186*t*

- Isocyanate adhesives
 polymeric 4,4'-diphenylmethane diisocyanate (pMDI), 75
 polymethylene diisocyanate, 75
 urethane bridge formation, 74–75, 75f
- J**
- Jute cellulose nano-fibrils (CNF)/hydroxypropylmethylcellulose nanocomposite, 341
- K**
- Kenaf/glass hybrid epoxy composite, 451–452, 452f
 Kenaf nanocellulose, 16–18, 18f
 Kyoto technique, 456
- L**
- Label driven optical biosensor, 227–229
 nanocellulose-based colorimetric biosensors, 231–233
 nanocellulose-based fluorescent biosensors, 230–231
- Label-free optical biosensor
 identification methods, 228
 nanocellulose-based label-free biosensors, 228–229
 Raman spectroscopy technique, 228
 recognition mechanisms, 227–228
 refractive index variations, 228
- Lactic acid, 80–81
- Laminated veneer lumber (LVL), 86
- Layer-by-layer spraying process, 225
- Leather-inspired nanocellulose-based films, 383–384
- Lemongrass essential oil, 322
- Level-off level of polymerization (LODP), 161–162
- Light-emitting diode (LED), 25–26
- Lignin, 42–43, 47, 50–51
 adhesive production, raw material for, 77–78
 building blocks of, 56, 56f
 definition, 55
 distribution of, 55–56
 formaldehyde crosslinking, 77–78, 78f
 nanolignin, 60–61
- Lignocellulosic biomass, 2–3, 50, 354, 356f
 for environmental remediation, 198–199
- Lignocellulosic biomaterials
 cellulose, 50 (*see also* Cellulose)
 chemical components of, 53f, 54
 chemical composition, 53–54
 component of, 53
 definition, 53
 extractive substances, 57–58
 hemicellulose, 50, 56–57
 lignin
 building blocks of, 56, 56f
 definition, 55
 distribution of, 56
 raw materials, 50
- Lignocellulosic fibers, 2, 376–377
- Linear low-density polyethylene (LLDPE), 290–291
- Lingocellulose nanofibers (LCNF), 87–89
- Liquid-assisted extrusion, 299–300
- Lithium-ion batteries, 496–497
- Lithium-ion battery (LIB) separator membrane, 459–460
- Low-density polyethylene (LDPE), 290–291, 291t
- Lung disease, 248–249
- M**
- Magnetic iron nanoparticles modified microfibrillated cellulose (FeNP/MFC), 421–425
- Magnetic nanocellulose aerogels, 426, 428
- Malachite green (MG) dyes, 427–428
- Malachite green isothiocyanate (MGITC), 229
- Malaysian GLP compliance program, 252
- Maleic anhydride (MA), 297–298
- Maleic anhydride grafted polypropylene (PPgMA), 302–303
- Mechanical fibrillation, 41
- Mechanical homogenization method, 485
- Mechanical refining, 12
- Medium-density fiberboard (MDF), 82, 83f, 85, 85t
- Melamine formaldehyde (MF) adhesive, 74
 condensation reaction, 73, 74f
 disadvantage, 73
 for exterior use boards, 73
- Melamine urea formaldehyde (MUF) adhesive, 73

- Melt compounding method, 327–330, 328*t*, 332
- PA11 and PA11/CNC nanocomposite film, 330–331, 331*f*
 - TPU/CNC nanocomposites, 332, 332*f*
- Melt processing, 108
- Mesothelioma, 248
- Metal-based nanocomposites, 90–95, 92–94*t*
- Metal-matrix composites, for automotive applications, 457–460
- Metal nanoparticles, 228–229
- Metal oxide gas sensor, 218–219
- Metal oxide/inorganic based nanocomposites, 100
- CdTe QDs, 101
 - cellulose nanofiber (CNF)-hydroxyapatite (HAP) composite foam, 101
 - copper oxide nanoparticles (CuO NPs), 99–100
 - magnetic nanoparticles, 100
 - mineral salts, 101
 - published studies, 95, 96–98*t*
 - quantum dots (QDs), 100–101
 - zinc oxide nanoparticle (ZnO NP), 99
- Metal oxides (MOx) chemical sensors, 220
- Methacryloxy silane, 182, 184*f*
- N,N'*-Methylene bisacrylamide (MBA), 144
- Methylene blue dye, 427–428
- Methylol phenol, 70
- Methyl paraben (MePRB), 221–222
- Microelectromechanical systems, 492–494
- Microfibrillated cellulose (MFC). *See* Cellulose nanofibers (CNF)
- Military appliances, nanocellulose in, 403–404
- Modulus of elasticity (MOE), 87–89
- Modulus of rupture (MOR), 87–89
- Multimetal nanoparticles, 221
- Multiwalled carbon nanotubes (MWCNTs), 226
- MXene nanosheets, 226
- N**
- Nanobentonite incorporated nanocellulose/chitosan aerogel, 421–425
- Nanocarbon based nanocomposites, 105–106
- applications, 101–105
 - composite films, 106
 - one-dimensional composites, 106
 - properties, 101–105
 - published studies, 101–105, 102–104*t*
 - single-walled and multiwalled CNTs, 106
 - three-dimensional nanocellulose/nanocarbon composites, 106–107
 - two-dimensional composites, 106
- Nanocellulose (NC), 2, 51–53, 58–60, 179–180
- acid hydrolysis, 440
 - adhesives, reinforcements in, 68–69
 - bioresins, 77–81
 - thermoplastic resins, 75–77
 - thermosetting resins, 69–75
 - aerogels, 201, 494 (*see* Aerogels)
 - applications, 52, 60, 107, 156, 169–170, 214, 376
 - biochemical applications, 160
 - biomedical applications (*see* Biomedical applications, nanocellulose in)
 - composite materials applications, 160
 - in food industry (*see* Food industry, nanocellulose in)
 - sensor applications (*see* Sensors)
 - surface coatings, 179
 - in textiles (*see* Textile industry, nanocellulose application in)
- automotive applications (*see* Automotive applications, nanocellulose)
- bacterial cellulose (BC) nanoribbons (*see* Bacterial nanocellulose (BNC))
- as bio adsorbent, 214–215
- as bioadsorbent, 410, 420–430
- from biological wastes, 14, 15*f*, 16*t*
- ginger, 15, 17*f*
 - kenaf, 16–18, 18*f*
 - sugarcane bagasse nanocellulose (SBCN), 19, 19*f*
 - sugar palm fiber (SPF), 10, 11*f*, 19–20, 20*f*
 - water hyacinth, 16, 18*f*
- bionanocomposites, applications of (*see* Bionanocomposites)
- biosynthesis process, 486
- bone repair and regeneration (*see* Bone tissue regeneration and repair)
- bottom-top approach, 200
- characteristics, 4–5, 5*t*, 214–215
- chemical pretreatment, 482
- chemical structure of, 2–3, 3*f*

- Nanocellulose (NC) (*Continued*)
commercial use, drawback of, 469–470
composites, reinforcements in (*see*
Composites, nanocellulose
reinforcement in)
cytotoxicity studies, 249–250, 251*t*
definition, 4–5, 156, 200, 440, 482
disadvantage, 339–340
in electronic applications (*see* Electronic
applications, nanocellulose in)
extraction, from cellulose fiber, 482, 483*f*
features, 4–5, 160, 397
functionalization, 417–418
heavy metals adsorption (*see* Heavy
metals, as chemical contaminants)
history of, 158–160
nanocrystalline cellulose (NCC) (*see*
Cellulose nanocrystals (CNC))
nanofibrillated cellulose (NFC) (*see*
Cellulose nanofibers (CNF))
nano-product, 397
natural fibers, pretreatment of, 9, 9*f*
acid hydrolysis, 9–10
alkali treatment, 10, 11*f*
deep eutectic solvents (DESs), 12
enzymatic pretreatment, 10–12
ionic liquids (IL), 12
mechanical refining, 12
organic solvents, 12
2,2,6,6-tetramethylpiperidine-1-oxyl
(TEMPO)-mediated oxidation, 10
preparation, from forest residues, 13–14,
13*f*
production of, 397
properties of, 6*f*, 27, 52, 58–60, 107,
156, 171, 179, 214–215, 354, 376,
470–471, 482, 487–488
crystallinity percentage/degree of
crystallinity, 5
degree of polymerization (DP), 6–7
extraction sources, 7, 8*t*
mechanical characteristics, 6–7
morphology, 6
thermal stability, 7
in pulp and paper industry (*see* Paper-
based products, nanocellulose in)
sources, 4–5, 59–60, 107, 160
top-bottom approach, 200
types of, 2–4, 3*f*, 214–215
- Nanocellulose aerogel foam/dodecylsulfate
(NAF/SDS), 428
Nanocellulose-carbon nanotube (CNT)-based
composite, 491
Nanocellulose-graphene matrix composites,
491
Nanocellulose/graphene oxide composites,
421–425
Nanocellulose/nanoalumina (NC/Al₂O₃)
aerogels, 429
Nanocellulose nanocomposites (NNC)
for automotive application
adoption, 446
biocomposites, 443–445
biodegradable materials, 444
bioplastics, 443–445
crystalline cellulose with other
reinforcements, 445, 446*t*
lightweight and carbon neutral, 447–448
nanomaterials, 445–446
polymers, 444
pulp, 447
research development and technological
innovation, 446–447
thermoplastic composites, 447–451, 449*f*
thermosetting composites, 450–451
in coating materials, 192
air resistance, 187–189, 189*f*
applications, 192, 193*f*
corrosion resistance, 189–190
crack resistance, 190, 191*f*
glass and aluminum substrates, 182
hemp fiber, 180
mechanical resistance properties,
185–187
polarized optical microscopy, 190–191,
191*f*
silane, surface modification with,
180–182, 181*f*
steel substrates, 182–184
water absorption properties, 189, 190*f*
water uptake and permeability, 191–192,
192*f*
electronic applications
conductive composites, 491–492
electrical properties, 491
lithium-ion batteries, separator of,
496–497
mechanical properties, 487–490

- organic field-effect transistors (OFETs), 496
- organic light-emitting diodes (OLEDs), 496
- piezoelectric and electroactive effect, 492–494
- supercapacitors (SC), 494, 495*t*
- thermal properties, 490–491
- thin film transistors (TFTs), 494–496
- in textiles (*see* Textile industry, nanocellulose application in)
- Nanocellulose/silica hybrid (CSH), 402
- Nanocrystalline cellulose (NCC). *See* Cellulose nanocrystals (CNC)
- Nanofibrillated cellulose (NFC). *See* Cellulose nanofibers (CNF)
- Nanofilms, 383–384
- Nanomaterials, 410, 417
- Nanoparticle-reinforced plywood panels, 87–89
- Nanosafety, of cellulose nanofiber, 245–246, 257
 - aquatic test system, 254–256
 - cellular test system, 249–250
 - industry and consumer products, application of, 247–249
 - mammalian test system, 250–254
 - physicochemical characterization, CNF suspension, 246
- Natural dyes, 411, 413*f*
- Natural fibers, 440, 460
 - components of, 439–440
 - definition, 9
 - lignocellulosic fibers, 2
 - polymeric materials reinforced with, 445
 - pretreatment of, 9–12, 9*f*
 - reinforced epoxy composites, 451–452, 452*f*
 - sources, 439–440
- Natural polymer nanocomposites, 469–470
- Nitrogen dioxide (NO₂) gas sensor, 216–217
- Nontoxic chemical sensor, 221
- Novolac type adhesives, 70–71
- O**
- Offset printing method, 380
- Oil palm biomass (OPB), 198–199, 315–316
 - chemical composition of, 316, 317*t*
 - CNF production, from OPMF (*see* Oil palm mesocarp fiber (OPMF), CNF production from)
 - oil palm empty fruit bunch (OPEFB), 316, 317*f*
 - oil palm fronds (OPF), 316
 - oil palm kernel shell (OPKS), 316, 317*f*
 - palm oil mill effluent (POME), 316, 317*f*
- Oil palm empty fruit bunch (OPEFB), 247, 316, 317*f*
- Oil palm fronds (OPF), 316
- Oil palm kernel shell (OPKS), 316, 317*f*
- Oil palm mesocarp fiber (OPMF), CNF
 - production from, 47
 - characterization analysis, 41
 - economic analysis, 41
 - economic evaluation, by SHS-WDM and KOH-WDM
 - capital expenditure (CAPEX), 45, 46*t*
 - components, 44–45, 44*t*
 - operating expenditure (OPEX), 45, 46*t*
 - profitability analysis, 45, 46*t*
 - hemicellulose removal, SHS and KOH pretreatments for, 40–41
 - mass balance, by SHS-WDM and KOH-WDM, 43, 43*f*
 - materials, 40
 - mechanical fibrillation, 41
 - SHS- and KOH-treated CNFs, properties of, 41–43, 42*t*, 43*f*
 - untreated, SHS- and KOH-treated pulps, properties of, 41–42, 42*t*
- Oil palm mesocarp fruit (OPMF), 316, 317*f*
- Oil-water separations, CNC aerogels for, 146–147
- Open circuit potential (OCP), 189–190
- Operational expenditure (OPEX), 41, 45, 46*t*
- Optical biosensors, 215
 - applications, 227
 - label-driven biosensors, 227–229
 - nanocellulose-based colorimetric biosensors, 231–233
 - nanocellulose-based fluorescent biosensors, 230–231
 - label-free optical biosensor
 - identification methods, 228
 - nanocellulose-based label-free biosensors, 228–229
 - Raman spectroscopy technique, 228
 - recognition mechanisms, 227–228
 - refractive index variations, 228

- Optoelectronic devices, 496
- Oral toxicity safety study, 252
- Organic aerogels, 141
- Organic field-effect transistors (OFETs), 496
- Organic light-emitting diodes (OLEDs), 25–26, 25*f*, 496
- Organic oils, 412, 428–430
- Organic pollutants, 410
- Organic solvents, 12
- Organic substances, 50, 57
- Organisation for Economic Co-operation and Development (OECD) test guidelines, 246, 249–256
- Organochlorines, 412, 414*t*
- Organophosphorus, 412, 414*t*
- Organosolv, 12
- Oriented strand board (OSB), 82–85, 83*f*, 84*t*
- Otolith/collagen-BC nanocomposites, 474–476
- Oxidative stress, 256, 257*f*
- P**
- Packaging, nanocellulose in
- antioxidants, 320–321
 - banana pseudostem fibers, nanofilm from, 384
 - biocomposite packaging film, 321–323
 - biodegradable guar gum/nano-crystalline films, 318
 - biodegradable polymers, 357–359, 360*t*
 - bio-nanocomposite films, 318
 - bionanocomposites, 16*t*, 21, 22*t*, 23–24*f*, 357–364, 370
 - biopolymers, 311–312
 - carrageenan, 313–315
 - cellulose (*see* Cellulose)
 - plasticizers, 319–320
 - starch, 312
 - cellulose, 359
 - films, 168–170
 - hydroxypropyl guar/cellulose-nanofibrils composite films, 318
 - natural nano-cellulose reinforced polymer nano-composite, 318
 - paper, 168–170, 383
 - petroleum-based packaging, 357
 - petroleum polymers, 311–312
 - plastic materials, 311–312
 - plastic packaging, 357–359
 - poly lactic acid (PLA), 168–169
 - polymer/CNF composites (*see* Polymer reinforced CNF composites)
 - polymer framework filler, 168–169
 - properties, 168
 - protective material for food, 357
 - surface modification, 168–169
 - synthetic packaging material, 311–312
 - tocopherol, 321
- Pad-dry-cure method, 402
- Palm cellulose ash, 219–220
- Palm oil mill effluent (POME), 316, 317*f*
- Paper-based NO₂ graphite electrode molecular sensor system, 218
- Paper-based products, nanocellulose in, 381, 388
- casting methods
 - aqueous casting solution, 378–379
 - nonaqueous casting solution, 378–380
 - challenges, 386–387
 - coating process, 380
 - filtration and papermaking processes, 381
 - nanofilm, 383–384
 - packaging paper, 383
 - paper sheet making, application in, 382–383
 - properties, 384–386
- Paraformaldehyde, 78
- Parallel-strand lumber (PSL), 86
- Particle-based wood composites
- oriented strand board (OSB), 82–85, 83*f*, 84*t*
 - particleboard, 82–83, 83*f*, 84*t*, 87–89
- Particleboard, 82–83, 83*f*, 87–89
- classification of, 83, 84*t*
 - definition, 83
 - disadvantage of, 83
- Passive cooling system, 482
- PEG-*block*-polyethylene (PEG-*b*-PE), 296
- Peptide-nanocellulose aerogel (PepNA), 146
- Percolating stiff nanoparticle network, 489
- Peroxidation process, 400–401
- Petrochemical-based plastics, 311–312
- Petroleum-based packaging, 357
- Petroleum polymers, 311–312
- 1,10-Phenanthroline (PHEN) sensor, 221
- Phenol-formaldehyde (PF) resin, 69, 87–89
- adhesives
 - disadvantages of, 71
 - features of, 70

- novolac type adhesives, 70–71
- preparation methods, 70–71
- resole adhesive, 70
- viscosity, solids content and gel time, increase in, 69
- alkaline and acidic condensation reaction, 70, 71*f*
- production of, 70
- Phenolic alcohol, 70
- Phenolic compounds, 58
- Phenol resorcinol formaldehyde (PRF) adhesive, 70, 74
- Phenols, 412, 414*t*
- Phosphoric acid hydrolysis, 329–331
- Phthalate plasticizers, 319
- Physisorption, 415–417
- Pickering emulsions, 165–166
- Piezoelectric nanocellulose/graphene-based sensors, 215
- Plant nutrients, 412, 414*t*
- Plasticizers, 319, 361–363
 - glycerol, 320, 320*t*
 - ionic liquid-like 1-allyl-3-methylimidazolium chloride, 328–329
 - natural plasticizers, 319
 - phthalates, 319
- Plastic materials, 311–312
- Plastic polymers, 357–359
- Plywood, 82, 83*f*, 86–89
- Polarized optical microscopy, 190–191, 191*f*
- Polyacrylamide (PAAM) hydrogels, 264, 268
- Polyacrylic acid (PAA) hydrogels, 264, 268
- Polyamide hot melt adhesives, 76
- Polyamides, 330–331, 331*f*, 446
- Polyaniline (PANi), 226
- Polybutylene adipate terephthalate (PBAT), 448–450
- Polycaprolactone (PCL) monomer, 298
- Poly-(diallyldimethylammonium chloride) (PDDAC), 233
- Polydimethylsiloxane (PDMS) composite, 225
- Polydimethylsiloxane (PDMS)/graphene (GR) hybrids, 221–222
- Polydopamine, 272
- Polyester fabric, 402
- Polyesters, 446
- Polyethylene (PE), 290–291, 291*t*, 327, 328*t*, 357–358, 448–450
- Poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate)(PEDOT-PSS), 225–226
- Polyethylene glycol (PEG), 100
- Poly(ethylene glycol) (PEG) hydrogels, 268
- Polyethylene-grafted-maleic anhydride (PEgMA), 297–298, 297*f*
- Polyethylene terephthalate (PET), 357–358
- Polyhedral oligomeric silsesquioxane (POSS), 402
- Polyhydroxyalkanoates (PHA), 294–295, 364
- Poly(3-hydroxybutyrate) (PHB), 294–295, 364
- Poly(3-hydroxybutyrate-co-3-hydroxyhexanoate) (PHBHHx), 294–295
- Poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV), 294–295, 330
- Poly(3-hydroxyhexanoate) (PHHx), 294–295
- Poly(3-hydroxyoctanoate) (PHO), 295
- Poly(itaconic acid/methacrylic acid)-grafted-nanocellulose/nanobentonite composite, 421–426
- Poly(lactic acid) (PLA), 168–169, 380
 - antioxidant active packaging, 323
 - cellulose nanocrystal (CNC), 330, 330*f*
 - cellulose nanofiber (CNF), 293–294
 - food packaging applications, 363
 - nanocomposites, 448–450
 - 3D printed composites, 489
- Poly(lauryl methacrylate)-*block*-poly(2-hydroxymethyl methacrylate) (PLMA-*b*-PHEMA), 296
- Polymer aerogels, 148
 - applications, 141, 142*f*
 - biopolymers/natural polymers, 140, 143
 - carbon/carbide-based aerogels, 141
 - cellulose and nanocellulose-based aerogels (*see* Cellulose, aerogels)
 - cross-linked chemical structure, 140, 141*f*
 - definition, 140
 - hybrid aerogels, 141
 - inorganic/organic aerogels, 141
 - macro and microscale, microporous structure, 140, 141*f*
 - synthesis, stages for, 141–142, 142*f*
 - synthetic polymers, 140–143

- Polymer composites/nanocomposites, 81–82, 107–109
- applications, 89
 - bio-nanocomposite films, 318
 - carbon-derived nanoparticles, 90
 - conductive nanomaterials, 90
 - hydroxypropyl guar/cellulose-nanofibrils composite films, 318
 - mechanical properties of, 487–490
 - melt processing, 108
 - metal-based nanocomposites, 90–95, 92–94*t*
 - metal oxide/inorganic based nanocomposites, 100
 - CdTe QDs, 101
 - cellulose nanofiber (CNF)-hydroxyapatite (HAP) composite foam, 101
 - copper oxide nanoparticles (CuO NPs), 99–100
 - magnetic nanoparticles, 100
 - mineral salts, 101
 - published studies, 95, 96–98*t*
 - quantum dots (QDs), 100–101
 - zinc oxide nanoparticle (ZnO NP), 99
 - nanocarbon based nanocomposites, 101–107, 102–104*t*
 - natural nano-cellulose reinforced polymer nano-composite, 318
 - problems of, 108
 - production techniques, 108
 - solution casting method, 108
 - thermally stable CNC reinforced with, 330, 332–333
 - polyamide (PA), 330–331, 331*f*
 - polylactic acid (PLA), 330, 330*f*
 - polypropylene (PP), 331–332
 - thermoplastic polyurethane (TPU), 332, 332*f*
- Polymer grafting strategy
- “grafting from” approach, 298
 - “grafting onto” approach, 296–298, 297*f*
- Polymeric 4,4'-diphenylmethane diisocyanate (pMDI), 75
- Polymer reinforced CNF composites, 290, 303
- fabrication methods, 298
 - compression molding, 302
 - extrusion, 298–300
 - injection molding, 301–302
 - internal melt-blending, 300–301, 300*f*
 - one-pot extrusion, 302–303
 - selection of, 298
 - solvent casting, 301
 - polyethylene (PE), 290–291, 291*t*
 - poly(hydroxyalkanoates) (PHA), 294–295
 - polylactic acid (PLA), 293–294
 - polypropylene (PP), 291–292, 292*t*
 - polystyrene (PS), 292–293
 - polytetrafluoroethylene (PTFE), 293
 - polyvinyl chloride (PVC), 293
 - surface modification strategies, 295–296
 - covalent modification, 296
 - noncovalent modification, 296
 - polymer grafting, 296–298, 297*f*
- Poly(methacrylic acid-*co*-maleic acid) grafted nanofibrillated cellulose (NFC-MAA-MA) aerogel, 421–425
- Polymethylene diisocyanate, 75
- Polyolefins, 446
- Polyphenols, 322
- in bearberry extract, 322–323
 - from green tea extract, 323
- Polypropylene (PP), 291–292, 292*t*, 448–450
- in food packing, 357–358
 - nanocomposites, 25
- Polysaccharide films, 383–384
- Polysaccharides, 269
- Polystyrene (PS), 292–293
- in food packing, 357–358
- Polytetrafluoroethylene (PTFE), 293
- Polyurethane BC nanocomposites, 473–474
- Polyurethane (PU) sponge sensor, 226
- Polyvinyl acetate (PVA) adhesives, 76–77, 77*f*
- Poly(vinyl alcohol) (PVA), 146–147, 225
- Polyvinyl alcohol (PVA)/CNF hydrogel, 224
- Poly(vinyl alcohol) (PVA) hydrogels, 268
- Polyvinyl alcohol (PVA) nanocomposites, 379, 448–450, 449*f*
- Polyvinylchloride (PVC), 293, 357–358
- Polyvinylidene chloride (PVdC), 168
- Porous materials
- characteristics, 139
 - polymeric materials
 - aerogels (*see* Aerogels)
 - applications, 139
- Potassium hydroxide (KOH) pretreatment, 40–42
- Pressure sensor, 492

Printing paper

- cellulose nanofiber, impact of, 368–369
- paper packaging market growth, 367
- paper strength, factors affecting, 367–368

Profitability analysis, 45, 46r

Protein-based edible film, 364

Q

Quantum dots (QDs), 100–101

Quartz glass microbalance (QCM), 215

R

Raman spectroscopy technique, 228

Reactive dyes, 401–402

Reactive POSS (R-POSS), 402

Refining, 368

Refractive index biosensing systems, 228

Regenerated cellulose nanofibers (RC), 440

Regenerated nanocellulose bioadsorbent, 420–421

Resin, 57

- bioresins, 77–81
- thermoplastic resins, 75–77
- thermosetting resins, 69–75

Resole type phenol formaldehyde adhesive, 70

Resorcinol adhesives, 69

Resorcinol formaldehyde (RF) adhesive, 74, 74f

Respirable airborne fibers, 248–249

Response surface methodology (RSM), 202–203, 363

Rhodamine B (RhB) dyes, 426

Rosemary extract, 322–323

S

Salt spray, 189–190

Seaweeds

- carrageenan (*see* Carrageenan)
- and cellulose, compatibility between, 318–319
- definition, 313

Self-healing stress sensors, 223–224

Sensors

- categories of, 214
- cellulose, 214
- functions, 214
- nanocellulose application in

acoustic wave-based sensor, 215

biosensors (*see* Biosensors)

challenges, 234–235

chemical sensor, 215, 219–223

future research, 235

gas sensor, 215–219

optical sensors, 215

piezoelectric nanocellulose/graphene-based sensors, 215

pressure sensor, 492

strain sensors, 223–226

temperature sensor, 492

nanomaterials, 214

role, 213

types, 214

Separation applications, CNC aerogels for, 146–147

Shake-flask method, 400–401

Sheet molding compound (SMC), 441–442

Silanization, of CNF, 296

Silica aerogels, 141–142

Silk fibroin hydrogels, 269–271

Silver-based nanocomposite, 91, 92–94r, 95

Silver nanoparticles (AgNPs), 219, 492

Silver nanoparticles–reduced graphene oxide (Ag/rGO) sheets, 399–400, 399r

Silver nanowires (AgNWS), 496

Silylated graphene oxide-grafted chemical-modified nanocellulose (Si-GO-g-CMNC), 222

Silylated NFC reinforced polypropylene (PP) composites, 490

Skin corrosion/irritation safety study, 253–254

Skin sensitization safety study, 253–254

Sodium alginate/gelatin hydrogels, 269

Sodium bicarbonate modified cellulose nanocrystal, 421–425

Sodium chlorite (NaClO₂), 40–41

Sodium dodecyl sulfate (SDS), 233

Sodium hydroxide (NaOH), 267

Sodium metaperiodate, 163–164

Sol-gel process, 141, 142f, 144, 147

Solution casting method, 108

Solvent casting method, 301, 327, 328r, 332, 332f

Soy protein adhesive, 79–80

Soy protein isolate-NCC film, 364

- Specialty papermaking, 354
nanocellulose biocomposites in
 food packaging applications (*see*
 Packaging, nanocellulose in)
 high durability paper, 364–367
 printing paper, 367–369
 popular companies, in production, 354,
 355*t*
- Spectrophotometers, 219
- Stabilizing agent, nanocellulose as, 165–166
- Starch, 312, 361–363
- Static cultivation method, 68
- Steel substrates, 182–184
- Strain sensors
 applications, 223
 electro-conductive hydrogels (ECHs),
 223–224
 nanocellulose strain sensor, 224–226
 piezoresistive and piezo capacitive
 properties, 223
 self-healing stress sensors, 223–224
 stretchable and conductive materials, 223
 stretchable strain sensors, 223–224
 technological shortcomings, 223
 uses, 223
- Strengths, weaknesses, opportunities, and
 threats (SWOT) analysis, 387
- Stretchable strain sensors, 223–224
- Succinic anhydride modified cellulose
 nanocrystal, 421–425
- Sugarcane bagasse nanocellulose (SBCN),
 19, 19*f*
- Sugar palm fiber (SPF), 10, 11*f*, 19–20, 20*f*
- Sugar palm nanocrystalline celluloses
 (SPNCs), 19–20, 20*f*
- Sulfonation, 91–95
- Sulphuric acid hydrolysis, 66, 162–163, 328,
 329*t*, 332
- Supercapacitors (SC), 494, 495*t*
- Supercritical drying, 141–142
- Superheated steam (SHS) pretreatment,
 40–42
- Superhydrophobic coating, 185–187
- Supermolecular aerogel (SA), 218, 226
- Suprathel, 343–344
- Surface enhanced Raman scattering (SERS),
 215, 228–229
- Surface-modified nanocellulose
 bioadsorbent, 420–421
- Surface plasmon resonance (SPR), 228–229
- Surfactant materials, 95
- Synthetic dyes, 411, 413*f*, 426
- Synthetic polymers, 140–143
- T**
- Tannin-based adhesives, 78–79, 79*f*
- Tea polyphenol-loaded chitosan
 nanoparticles, 322
- Temperature sensor, 492
- TEMPO-oxidized cellulose nanofiber
 (TOCN), 490–491
- TEMPO oxidized-cellulose nanofibrils
 (TOCNFs)-gold nanoparticles,
 427–428, 427*f*
- Tensile index, 187, 188*f*
- Tetracycline hydrochloride, 272
- 2,2,6,6-Tetramethylpiperidine-1-oxyl
 (TEMPO)-mediated oxidation, 6–7,
 10, 64, 161–164, 267, 377–378
- Textile industry, nanocellulose application in,
 397, 404–406
 challenges, 404
 features, 398
 functionalized nanocellulose
 antimicrobial properties, 400–401, 401*t*
 dye, adsorption kinetics of, 401–402
 goals, 398
 grafting process, 398
 surface modification, chemical
 functionalities, 398
 thermal insulation properties, 398–400,
 399*t*
 textile-related industry
 medical tools, 402–403
 military sector, 403–404
- Thailand GLP Monitoring Programme, 253
- Thermal conductivity (TC), of nanocellulose,
 398–400, 399*t*
- Thermal management, of electronic material
 devices, 482
- Thermogravimetric analysis (TGA), 19–20
- Thermogravimetry analyzer, 41
- Thermoplastic composites, for automotive
 applications, 447–450, 449*f*
- Thermoplastic polyurethane (TPU), 332,
 332*f*
- Thermoplastic polyurethane (TPU)
 membranes, 226

- Thermoplastic resins
automotive application, 448
hot melt adhesives
 applications, 75
 characteristics, 75
 ethylene-vinyl acetate (EVA)
 copolymer, 75–76, 76f
 features, 75
 high viscosity, 75–76
 polyamide adhesives, 76
 polyvinyl acetate (PVA) adhesives, 76–77, 77f
- Thermoplastic starch, 361–363
- Thermosetting resins
aminoplastic adhesives, 69
condensed adhesives, 69
formaldehyde, 69–70
isocyanate adhesives, 74–75, 75f
melamine formaldehyde (MF) adhesive, 73–74, 74f
melamine urea formaldehyde (MUF) adhesive, 73
phenolic resins (*see* Phenol-formaldehyde (PF) resin)
phenol resorcinol formaldehyde (PRF) adhesive, 74
resorcinol formaldehyde (RF) adhesive, 74, 74f
urea formaldehyde (UF) adhesives (*see* Urea formaldehyde (UF) adhesives)
- Thickness swelling (TS), 87–89
- Thin film transistors (TFTs), 494–496
- 2-Thiobarbituric acid-reactive substances (TBARS), 322
- Thiolated-SNC, 421–425
- Three-dimensional (3D) printing, 270–271, 340–341, 474, 475f
- TiB₂-reinforced Ti matrix composite, 457
- TiO₂ nanoparticles, 417
- Tissue engineering (TE), 341–342
 for bone repair and regeneration, 469–476, 473f, 477t
 nanocellulose hydrogels, 270–271
- Titanium oxide (TiO₂), 217
- Tocopherol, 321
- Tooth-mixing element (TME) screw, 302–303, 302f
- Transmission electron microscopy (TEM), 19–20
- Tricyanofuran-hydrazone (TCFH), 232–233, 232f
- Twin-screw extrusion technique, 25
- ## U
- Ultra-high molecular weight polyethylene (UHMWPE), 290–291
- Ultrasonication, 329, 329t
- Ultrasonic pulse-echo process, 225
- Urea biosensor, 232
- Urea formaldehyde (UF) adhesives, 69, 71–73, 87–89
 advantages, 71
 cellulose nanocrystals (CNC), interaction with, 87–89
 cellulose nanofibers (CNF), 87
 curing in, 72
 formaldehyde emission, 72–73
 hardening reaction, 72–73
 for interior use, 72
 particleboard panels bonded with, 87
 polymeric condensation reaction, 71–72, 73f
 powder, 72
 production of, 72
 structure, 72–73
 synthesis, 72, 73f
- ## V
- Veneer-based wood composites, 85
 glue-laminated timber (Glulam), 86–89
 laminated veneer lumber (LVL), 86
 parallel-strand lumber (PSL), 86
 plywood, 82, 83f, 86–89
- Vinyl acetate, 76
- Viscoelastic polymers, 223
- Vitamin E. *See* Tocopherol
- ## W
- Waste sulfide spent liquor (SSL), 77–78
- Wastewater remediation. *See* Water contaminants
- Water contaminants
 adsorption process, 415–417
 classes, 413–414
 purification procedures, 413–414
- Water hyacinth nanocellulose, 16, 18f
- Water-soluble polymer, 448
- Weather sensors, 234

- Wet disk milling (WDM), CNF production
mechanical fibrillation, 41
SHS- and KOH-treated CNFs, properties
of, 41–43, 42*t*, 43*f*
SHS-WDM and KOH-WDM methods
economic evaluation, 44–45, 44*t*
mass balance, 43, 43*f*
- Wet-spinning method, 106–107
- Wood-based composites, 81–82
component of, 82
disadvantages of, 82–83
general elements in, 82, 82*f*
medium-density fiberboard (MDF), 82,
83*f*, 85, 85*t*
particle-based wood composites
oriented strand board (OSB), 82–85,
83*f*, 84*t*
particleboard, 82–83, 83*f*, 84*t*, 87–89
veneer-based wood composites, 85
glue-laminated timber (Glulam), 86–89
laminated veneer lumber (LVL), 86
parallel-strand lumber (PSL), 86
plywood, 82, 83*f*, 86–89
- Wound dressing applications, 337–338,
342–343
animal-derived nanocellulose, 344
antimicrobial substances, 344, 345–346*t*
bacterial nanocellulose (BNC), 343,
402–403
hydrogels, 271–272
plant-derived nanocellulose,
343–344
textile used for, 400
- X**
- X-ray diffraction (XRD), 19–20
- Y**
- Young's modulus, 182, 183*t*
- Z**
- Zebrafish (*Dania rerio*), 254–256, 257*f*
- Zeolitic imidazolate framework-67 (ZIF-67)
modified bacterial cellulose/chitosan
(BC/CH) composite aerogel, 427–428
- Zinc oxide (ZnO)
functionalized ZnO/CNC, bacteriostatic
effect of, 400–401
nanomaterials, 220
nanowire sensor, 218
plasma treatment, 401
- Zinc oxide nanoparticle (ZnO NP), 96–98*t*,
99