

## Review

## Nanocellulose in food packaging: A review

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## ABSTRACT

The research in eco-friendly and sustainable materials for packaging applications with enhanced barrier, thermo-mechanical, rheological and anti-bacterial properties has accelerated in the last decade. Last decade has witnessed immense interest in employing nanocellulose (NC) as a sustainable and biodegradable alternative to the current synthetic packaging barrier films. This review article gathers the research information on NC as a choice for food packaging material. It reviews on the employment of NC and its various forms including its chemo-physical treatments into bio/polymers and its impact on the performance of nanocomposites for food packaging application. The review reveals the fact that the research trends towards NC based materials are quite promising for Active Packaging (AP) applications, including the Controlled Release Packaging (CRP) and Responsive Packaging (RP). Finally, it summarizes with the challenges of sustainable packaging, gray areas that need an improvement/focus in order to commercially exploit this wonderful material for packaging application.

## 1. Introduction

Whether be it the prevention of physical damage, external contamination or deterioration, packaging is necessary for the protection of goods (Wang & Wang, 2017). Effective packaging apart from physical protection offers conservation of the quality and safety of the food during transportation and storage. The shelf life of the food product can be extended chiefly by preventing- i) permeation of moisture, various gases including oxygen, carbon dioxide, ii) exposure to light, iii) deterioration by micro-organisms. Traditional materials like glass, aluminum, tin, petro-based polymers impart requisite strength and barrier properties but offers economic and ecological setbacks.

There has been an increase in demand for packaging materials which are non-petroleum based, biodegradable, pose very minimal threat to environment and are manufactured from sustainable and renewable resources (Han, Yu, & Wang, 2018). Burgos et al. presented the need of biodegradable polymers to replace the petro-based synthetic polymers as raw materials for wrapping (Burgos, Tolaguera, Fiori, & Jiménez, 2014). Conventionally used synthetic polymers have an edge over the biopolymers. Biopolymers have poor barrier, thermal and mechanical properties compared to their synthetic counterparts (Jabeen, Majid, & Nayik, 2015). This limits their usage in commercial applications such as food packaging. Partial solution to these concerns is crosslinking, grafting or functionalization of the monomers (Hambardzumyan et al.,

2015). poly(lactic acid) (PLA) (Ahmed & Varshney, 2011), poly-hydroxyalkanoates (PHAs) (Panaitescu, Frone, & Chiulan, 2016; Yu, Yan, & Yao, 2014), chitosan (El Miri et al., 2015), proteins obtained from animals (gelatin, caseinates, etc.) and vegetables (soy, gluten, etc.) are among the widely studied biodegradable and bio-based polymers for food packaging (Peelman et al., 2015). It is possible to improve the chemical and structural properties of biopolymers by addition of reinforcing agents, making them eligible for commercial applications.

Recent years have seen increased attraction towards the nano-technological applications in packaging (Huang, Li, & Zhou, 2015). Nanofillers are fillers or additives with their size lying in the range of 100 nm (Poole & Owens, 2003). These nanofillers are used in improving the properties of polymeric material in a composite for desired applications. Different thin composite films produced using nanofillers have vast applications in coating, packaging, as they provide impermeability towards moisture, gases such as oxygen, carbon dioxide and aromas, etc. and have good antibacterial properties as well. Such properties of the films ensure the packed foods last long. Nanocellulose (NC), prepared by breaking down cellulose fibers, is one such biodegradable, renewable nanofiller (biopolymer) that produces low carbon footprint. Plants create around 75 billion tons of cellulose every year, making this an extremely abundant material (Feng et al., 2018). For cellulose derived packaging, three types of cellulose are used, namely, cellulose nanocrystals (CNC), cellulose nanofibrils (CNF), and bacterial nanocellulose

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(BNC). Table 1 shows the comparative properties of these cellulose types. The details of production of NC including its pretreatment and isolation, technologies for production is not the part of our discussion and is elaborated elsewhere (Abdul Khalil et al., 2016; Ferrer, Pal, & Hubbe, 2017). Currently, CNC and CNF are incorporated as reinforcing agents into various biopolymers for making green composites (Moustafa, Youssef, Darwish, & Abou-Kandil, 2019). In recent past, these nanomaterials have been increasingly employed in packaging applications. NC can synergistically work with other materials to enhance barrier, thermo-mechanical, and rheological properties of the nanocomposite. Intermolecular and intramolecular hydrogen bonding makes cellulose insoluble in almost all solvents as these bonds impart high strength to it. Research has also commenced towards the recycling of this NC (Shanmugam, Doosthosseini, Varanasi, Garnier, & Batchelor, 2019) (Table 2) and employing waste paper as a source to extract it further (Kumar, Pathak, & Bhardwaj, 2020). It has been reported that the films produced from NC can be voluntarily recycled and reprocessed into a packaging film without drastically degrading the properties of the same (Shanmugam et al., 2019). The foremost reason behind this would be its carbon neutrality, non-toxic nature, recyclability and sustainability (Vilarinho, Sanches Silva, Vaz, & Farinha, 2018). Attributed to the distinctive properties of NC, tunable surface chemistry, barrier

properties, mechanical strength, crystallinity, biodegradability and non-toxicity, high aspect ratio, it is an arising credible renewable green substrate for food packaging applications (Ferrer et al., 2017; Lacroix, Criado, Frascini, & Salmieri, 2014). Fig. 1 shows the number of SCI indexed journals published employing NC in packaging applications. Number of patents on NC granted demonstrates its overall growing importance in coming future (SciFinder Scholar database, Nov. 2020). Research in the production of eco-friendly bio-nanocomposites for application in the food packaging sector has stimulated recently (Berthet, Angellier-Coussy, Guillard, & Gontard, 2016; Criado et al., 2014; Dhar, Bhardwaj, Kumar, & Katiyar, 2015; Hambardzumyan et al., 2015; Sanchez-Garcia, Lopez-Rubio, & Lagaron, 2010; Takala et al., 2013). Such composite films have also been made and tested for applications including intelligent packaging, (Abdul Khalil et al., 2016) see-through packaging (Azeredo, Rosa, & Mattoso, 2017), UV screening packaging (Boufi et al., 2016), antimicrobial packaging (Osong, Norgren, & Engstrand, 2016), etc. using different bio-polymeric matrices such as agar (Reddy & Rhim, 2014; Shankar & Rhim, 2016), chitosan (Dehnad, Mirzaei, Emam-Djomeh, Jafari, & Dadashi, 2014), carboxymethyl cellulose (CMC) (Benyoussef et al., 2015), polyvinyl alcohol (PVA) (Ching, Rahman, Ching, Sukiman, & Cheng, 2015; Lani, Ngadi, Johari, & Jusoh, 2014). The NC composites are a green option for manufacturing packaging materials (Sharma, Thakur, Bhattacharya, Mandal, & Goswami, 2019). The plenty of hydroxyl groups present on the surface of NC allows its chemical crosslinking with different polymers and thereby strengthen

**Table 1**  
Comparative properties of various NC including CNC, CNF, and BNC.

Properties	CNC	CNF	BNC	References
	L:	L:	L: 100 -	
Dimensions (nm)	100-400	700-2000	>1000	
	D: 3-40	D: 6-80	D: 30-60	
Elastic Modulus (GPa)	130-250	20-60	15-138	(Rajinipriya, Nagalakshmaiah, Robert, & Elkoun, 2018); (Kargarzadeh et al., 2018); (Rol, Belgacem, Gandini, & Bras, 2019); (Klemm et al., 2018)
Tensile Strength (MPa)	7500-7700	350-500	20-2000	
Thermal stability up to (°C)	220	260	300	
Crystallinity (%)	60 to 90	40-70	50-60	
Transparency (%)	> 90	80-90	> 90	

**Table 2**  
Recent research in improving the water vapor barrier properties.

Composite	Concentration	WVTR (g m <sup>-1</sup> s <sup>-1</sup> Pa <sup>-1</sup> ) x10 <sup>11</sup>	Authors
<b>Cellulose as matrix phase</b>			
Unused NC film		5.45	(Shanmugam et al., 2019)
Recycled NC film		12.90	(Maliha et al., 2020)
Bismuth complex / MFC sheet	Bismuth Complex - 0.1-2 wt.%	3 - 3.5	
<b>Cellulose as reinforcing phase</b>			
CNC incorporated in PLA		1.98	(Khodayari et al., 2019)
with PEE and TBE as additives (1 wt.% of CNC)	1 v/v% PEE 1 v/v% TBE	2.00 2.06	
Chitosan crosslinked with CMC as matrix and CNC as reinforcing phase	0 wt.% 5 wt.% 10 wt.%	0.15 0.13 0.09	(Chi & Catchmark, 2018)
C-CNC whiskers in Cassia-gum	0 wt.% 4 wt.% 6 wt.%	34.66 25.05 26.12	(Cao et al., 2020)
	PVOH - 0 wt.% Glycerol - 0 wt.% PVOH - 2.5 wt.%	2.38 1.87	
Bacterial Cellulose (BC)-PVA-Glycerol films with varying concentration of the PVA and Glycerol	Glycerol - 0 wt.% PVOH - 0 wt.% Glycerol - 2.5 wt.% PVOH - 2.5 wt.% Glycerol - 2.5 wt.%	5.92 17.20	(Cazon et al., 2020)
MCC incorporated in PVA with AgNPs	MCC - 16 wt.% AgNPs - 5 wt.% MCC - 16 wt.%	37.39 g/m <sup>2</sup> h 14.36 g/m <sup>2</sup> h	(Sarwar et al., 2018)
Alginate and CNF complex prepared by blending	CNF - 9 wt.%	26.70	(Zhao et al., 2020)
Prepared by layer by layer formation	CNF - 9 wt.%	26.10	
Chitosan and CNF composite Prepared by blending	CNF - 9 wt.%	196.70	
Prepared by layer by layer formation	CNF - 9 wt.%	28.90	
CNC extracted from Bamboo incorporated into Corn starch matrix	4 wt.%	4.25	(Chen et al., 2019)

the composites further.

This review throws light on the advancement of NC reinforced bio/polymer nanocomposites for packaging application. It emphasizes on the property enhancement like impermeability of various gases and water vapor, transparency; improving resistance towards UV light, oxidants, micro-organisms, etc. of such nanocomposite films.

## 2. Gas barrier properties

Oxygen barrier is important in food packaging as its presence assists the aerobic microorganisms to spoil the food and lose its nutritional properties. In order to extend the shelf-life of the food, it is required to maintain low oxygen environment. NC has an ability to form hydrogen bonds (with itself as well as with other biopolymers) that forms a dense network. This impedes various gas molecules to pass through due to small pore size. Apart from that, its high crystalline regions are impermeable to gases, making it a good candidate to impart gas barrier properties. Permeability of gas depends upon the dissolution of gas and its diffusion rate in the packaged film (Wang, Gardner et al., 2018). NC increases the diffusion path length of the gases (or water vapor) that permeate the packaging as shown in Fig. 2 (Nair, Zhu, Deng, & Ragauskas, 2014). It is worth noting that, up to a relative humidity (RH) level of about 70 %, the hydrogen bonding reduces the gas permeability because of the increase in cohesion (Mascheroni et al., 2016; Rampazzo et al., 2017; Sukyai et al., 2018). As the RH value increases further, the

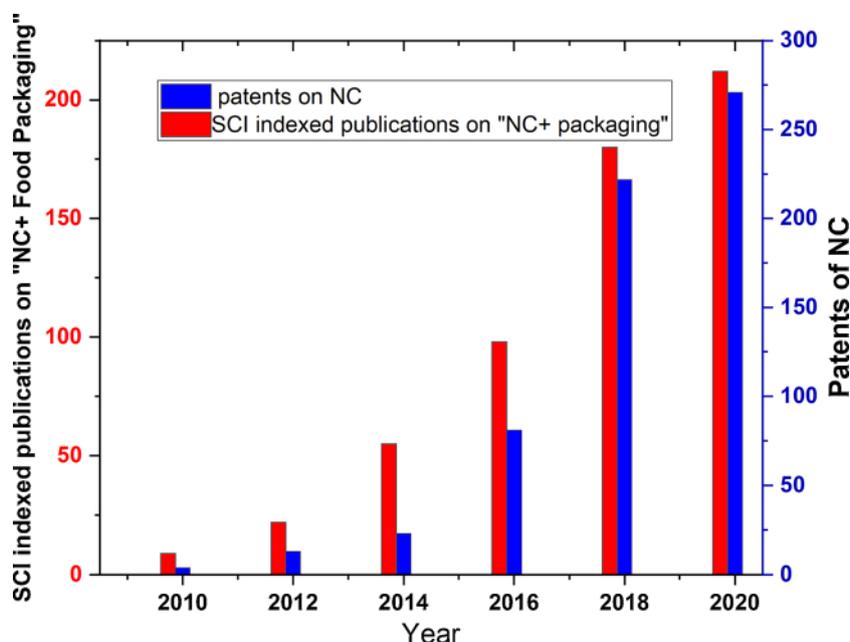


Fig. 1. Growing importance of NC reflects in increasing the number of SCI indexed publications and patents granted over the last decade.

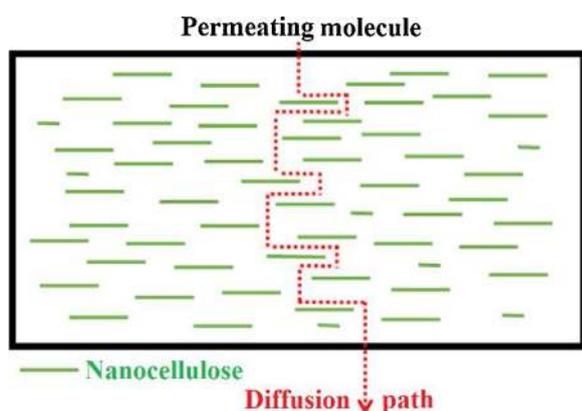


Fig. 2. Diagram showing the increase in diffusion path of the permeating molecule due to the presence of NC (Nair et al., 2014).

moisture (and hence gases too) in the surroundings, begins to diffuse into the polymer. Thus, it can be inferred that the atmospheric conditions need to be taken into consideration while designing the packaging material.

Improved barrier properties with respect to packaging of food requiring less moisture content for instance nuts, dried fruits, and spices, etc. have been obtained through multilayer packaging. CNFs were layered on polyethylene terephthalate (PET) and the resulting film was further coated with low-density polyethylene (LDPE) via extrusion, giving a three-layered structure (Vartiainen, Kajjunen, Nykanen, Maim, & Tammelin, 2014). The barrier performance of this multilayered film was better than the commercial ethylene vinyl alcohol multi-layered barrier film. With increasing grammage of CNF (from 1 to 7 g/m<sup>2</sup>) coating on paper, the air permeance and oxygen permeability (OP) of the paper was reduced by almost 70 % and 10<sup>4</sup> times respectively (Aulin, Gällstedt, & Lindström, 2010; Lavoine, Desloges, Khelifi, & Bras, 2014). Coating on a paper is important to increase the barrier properties and moisture resistance. Addition of CNF coating also increased tensile strength and oil resistance. Missio et al. (Missio et al., 2018), in their work, used condensed tannin and CNFs, where CNFs contributed to the mechanical properties and tannin served as an antioxidant (Duval &

Avérous, 2016). Antioxidants mitigate the oxidation kinetics of the food, even in presence of oxygen. The prepared composite had better surface hydrophobicity as well as high density resulting in an excellent 6-fold increase in barrier properties towards air when compared to a pure CNF film (19.27 mL/min) and 90-folds when compared to a regular A4 size paper (277.2 mL/min). The resistance towards air was about 3.1 mL/min, which is close to that of plastic packaging for chocolate made from poly-propylene. The film produced also manages to overcome the shortcoming of NC film, wherein the density of the film needs to be compromised in order to decrease the affinity towards water, thereby degrading the barrier properties (Hubbe et al., 2017).

Earlier, Belbekhouche et al. reported that the gas permeability in CNF films was lesser than in CNC films (Belbekhouche et al., 2011). Formation of dense entanglements by the CNF network films was attributed to lesser permeation of gases through it. It was observed that the CNF films displayed Oxygen Transmission Rate (OTR) of approximately 17 mL/m<sup>2</sup>/day (Syverud & Stenius, 2009) (see Fig. 5), which is equivalent to many petro-based polymers (Parry, 1993). Mondragon et al. fabricated a film involving gelatin matrix and reinforcing phase of CNFs and CNCs separately. The OTR through gelatin was reduced by 21 % and 36 % at 5 and 10 wt.% concentration of NC (similar effects were observed employing CNC and CNFs), respectively (Mondragon, Peña-Rodríguez, González, Eceiza, & Arbelaiz, 2015). However, Higher is the crystallinity of the film; higher is the gas barrier property. Compared to other types of NC (CNFs or BNCs), CNCs have highest degree of crystallinity, providing better barrier to gas molecules. Dhar et al. reported that just 2 % CNC in poly(3-hydroxybutyrate) (PHB) reduced the oxygen permeability of PHB films by 65 % (Dhar et al., 2015). The improvement in barrier properties is dependent on the concentration of CNC. It is preferential to incorporate the CNCs into the bulk of the polymer to obstruct the diffusion of gases through the composite (Karkhanis, Stark, Matuana, & Sabo, 2018; Luzi et al., 2016). CNC has proven its worth as an oxygen barrier material, when incorporated into kappa carrageenan (Azizi and Mohamad, 2018), collagen (Long et al., 2018), poly(propylene carbonate) (PPC) with polyethylene glycol (PEG), (Jiang, Zhang, Feng, Zhang, & Wang, 2017) poly(L-lactic acid) (PLLA) (Kakroodi, Kazemi, Nofar, & Park, 2017), CNC has also shown great impermeability towards oxygen as a coating on PET (Rampazzo et al., 2017). Despite of the fact that, high crystallinity of CNCs results in least OTR, the films or coatings prepared from CNFs are

less brittle than that prepared from CNCs.

The property of NC of being tortuous, which in turn reduces the permeation of the gas molecules through composite films, is worth studying in future (Ferrer et al., 2017). The free spaces between the molecules, and the cohesive energy density of the same also determine the gas permeability of a material (Miller & Krochta, 1997). When CNCs are incorporated into a matrix, high cohesion energy density is observed due to the presence of a network of hydrogen bonds. Also, the free volume is less and the chain mobility is reduced attributed to the presence of nanoparticles (Lagaron, Catalá, & Gavara, 2004; Sharma & Dhanjal, 2016). Fig. 3 displays how the oxygen barrier properties (OBP) of poly(L-lactic acid) (PLLA) vary with addition of functionalized CNC (Li, Bao et al., 2019). The oxygen permeability of neat PLLA was reduced by 66.4 % on the addition of PEG (1 g) and CNC (0.5 wt.%). (2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO)-oxidized CNCs have also been testified to act as oxygen barriers (Vähä-Nissi et al., 2017). Tyagi et al. prepared a film, a composite system of CNC and CNF, which decreased the OTR by a factor of around 260 and air resistance increased by a factor of 300, relative to an uncoated paper (Tyagi, Lucia, Hubbe, & Pal, 2019). Also, the increase in grease and oil resistance was analogous to that of the fluorochemicals. Fluorochemicals, because of their credible lipophobic and hydrophobic properties, are a good raw material for waterproof and non-stick food packaging (Schaidler et al., 2017).

### 3. Water vapor barrier properties

The water vapor (WV) permeation into the film is an important concern as it directly affects the transmission of oxygen through the film. It has been reported that, at a relative humidity level of 80 %, the OTR was increased 20 times (Cozzolino, Campanella, Türe, Olsson, & Farris, 2016). The presence of water plasticizes the film. The gas diffusion rate upsurges as the water molecules weaken the film cohesion at higher humidity levels (Lagaron et al., 2004). Factors that affect the water vapor transmission rate (WVTR) of a packaged polymer/composite film are pressure, temperature, crystallinity (the diffusion and sorption predominantly take place in the amorphous region of a polymer) and hydrophilicity (polysaccharides are hydrophilic in nature), film density and thickness, pore size and structure. For a film to increase the shelf life of a food product, it needs to avoid the moisture interaction during the film handling, transportation and application. Research is exhaustively being carried out employing NC either in coating, or as a filler in a

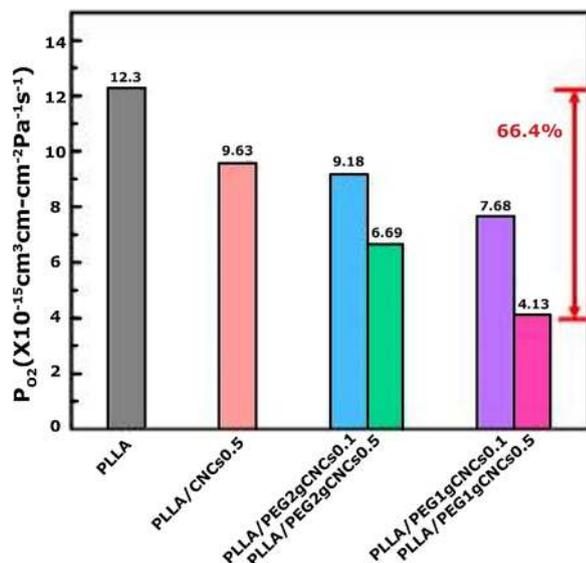


Fig. 3. Oxygen barrier properties of CNC (functionalized) with Dopamine induced polyethylene glycol incorporated in poly(L-lactic acid) biocomposite. (L. Li et al., 2019).

composite film or as a matrix alone.

#### 3.1. Films based on Cellulose Nanofibrils

The barrier properties of NC films are good for gases but are very poor for WV when used in its unmodified form. The WVTR of CNF film was found approximately  $174 \text{ g/m}^2/\text{day}$  (Rodionova, Lenes, Eriksen, & Gregersen, 2011) and is very high compared with LDPE ( $\sim 15\text{--}20 \text{ g/m}^2/\text{day}$ ) (Parry, 1993). Consistent efforts are on in improving water vapor barrier properties (WVBP). Sharma et al. heated the CNF films at  $175^\circ\text{C}$  for 3 h and observed 50 % reduction in WV permeability (Sharma et al., 2014). Heating caused an increase in the crystallinity and hydrophobicity of CNF and reduction in porosity that hinders the diffusion of water molecules. Modified CNF, having high specific area and aspect ratio, gives entanglement ability with the network (B. Wu et al., 2017). However, TEMPO oxidized CNF (TOCN) falls short when talked about its water barrier properties ( $\sim 235 \text{ g/m}^2/\text{day}$  for 50 % RH), even though its performance as a barrier against gases is comparable to synthetic polymers (See Fig. 5). Bideau et al. synthesized TOCN, PVA and Polypyrrole (PPy) nanocomposite films by two ways. i) *in-situ* by chemical polymerization (Bideau, Bras, Saini, Daneault, & Loranger, 2016) and also ii), distributed pyrrole over this TOCN/PVA film and allowed it to polymerize (Bideau, Bras, Adoui, Loranger, & Daneault, 2017). In the second processing case, the performance of the film was tested against gas and WVBP. PPy, being hydrophobic and at the outer layer, protects the film from water permeation ( $18 \text{ g/m}^2/\text{day}$ ). This film's performance was similar to the commercially available petro-based polymers. ( $16.8 \text{ g/m}^2/\text{day}$ ). In the first case, it was observed through EDX studies that PPy located on the composite surface, resulting in increased contact angle from  $54.5$  to  $83^\circ$  and corresponding increased heat protection. The performance of this film was evaluated against food containing bacteria (*B. Subtilis* and *EscherichiaColi*). It was demonstrated that by slight leaching of PPy, this film acted as an active packaging on meat and killed the bacteria by contact.

Recently, research is being focused on preparing a coating of NC onto biopolymers, thereby making the packaging completely biodegradable. NC being dispersible, an aqueous coating can be applied as a pure thin layer or as a composite with other commonly used materials. CNFs has also been used as an edible coating on fruits, for instance, coating of aqueous slurries of CNF and calcium carbonate nanoparticles onto blueberries (Zhao, Simonsen, Cavender, Jung, & Fuchigami, 2014), or coating on strawberries with a mixture of 1 % chitosan and 5 % NC (Dong, Li, Liu, & Zhu, 2015). These coatings proved to be useful in reducing the leakage of anthocyanins, weight loss and decay rates. The use of paperboards for packaging, aims at extending the shelf life of the packaged food while reducing the packaging waste (Silvestre, Duraccio, & Cimmino, 2011). However, the use of paperboards alone for food packaging is not desirable due to its weak mechanical properties and its susceptibility to humidity (Biedermann-Brem, Biedermann, & Grob, 2016). The emergence of paperboards as a substrate for protective coatings was reviewed by Park (Park, 2005). Water vapor permeation rate can possibly be reduced via making a multi-layered system, which would protect the substance inside from the effects of moisture (Schade, Weinkoetz, & Assmann, 2015; Jari Vartiainen et al., 2016) over a substrate. One such multi-layered packaging was formulated by Koppolu et al. (Koppolu et al., 2019). The authors developed a thin multilayer coating of NC reinforced PLA over a paperboard via slot-die coating and extrusion coating (Fig. 4(a)) (Koppolu et al., 2018; Kumar, Elfving, Koivula, Bousfield, & Toivakka, 2016). It lowered WVTR by 23 % even at 90 % humidity, at  $38^\circ\text{C}$ , reduced OTR and heptane transmission rate by 98 % and 99 %, compared to PLA-coated paperboard and the paperboard alone respectively. PLA compensated for the poor water vapor resistance of NC. In their work, Bideau et al. emphasized on the use of PPy and TOCN coating. The coating was tested and compared with an uncoated paperboard by storing cherry tomatoes for 10 days (Fig. 4(b)). The tomatoes kept inside the coated paperboard retained

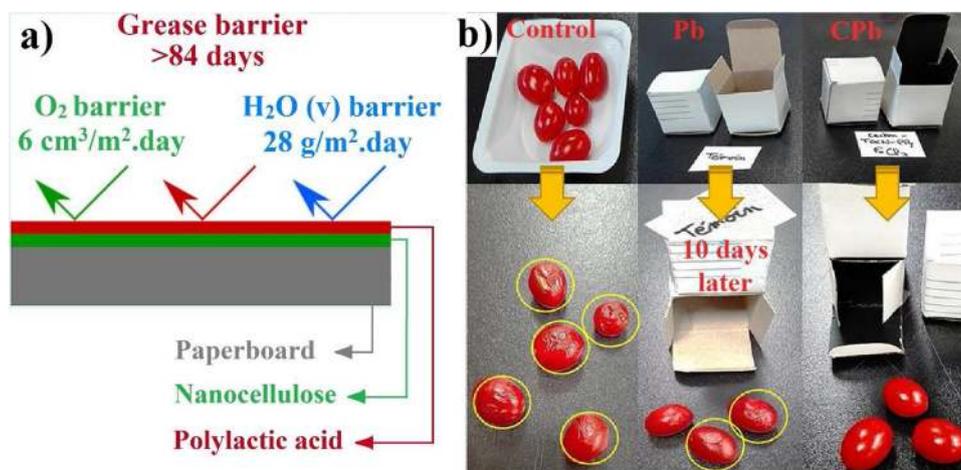


Fig. 4. (a) A diagrammatic representation of multilayered packaging system restricting grease, O<sub>2</sub> and H<sub>2</sub>O (reproduced from (Koppolu et al., 2019)) (b) Packaging simulation of Cherry tomatoes in coated and uncoated paperboard for 10 days. (Reproduced from (Bideau et al., 2018)).

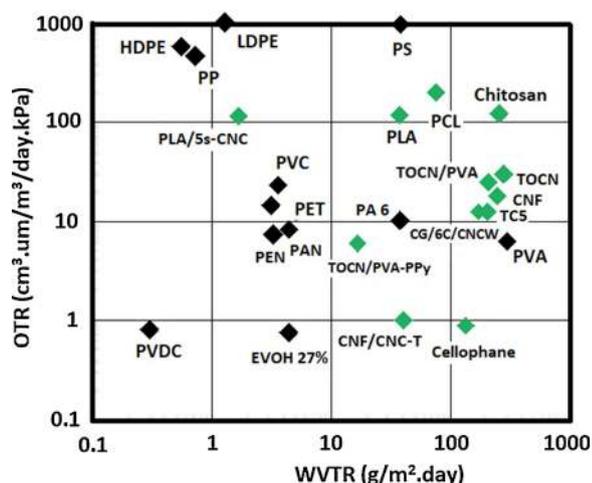


Fig. 5. WVTR and Oxygen transmission rate (OTR) Synthetic polymers compared to that of the Biopolymers and composite packaging films. (Bardet et al., 2015; Cao et al., 2020; Fortunati, Peltzer, et al., 2012b; Ma et al., 2018). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article). Adapted from (Bideau et al., 2017) (Dark Points – Petro-based Polymers, Green Points - Biopolymers).

their texture and stayed firm. The dense network formed by the combination of TOCN and PPy, providing a barrier against moisture and oxygen, was responsible for the same. The authors also suggested that the 80 % of the paperboard could be reused by removing the layer of PPy. (Bideau, Loranger, & Daneault, 2018)

### 3.2. Films based on Cellulose Nanocrystals

Similar to CNFs, CNCs have been employed as coatings for packaging application. Azeredo et al. compared the reinforcing ability of CNC derived from coconut fiber on alginate-acerola puree-based coatings (Azeredo, Miranda, Ribeiro, Rosa, & Nascimento, 2012). A test was carried out over a period of 7 days by comparing the properties of acerola before and after this interval. The CNC reinforced film decreased the Water Vapor Permeability (WVP), fruit weight loss, ascorbic acid loss, and decay incidence by 30 %, 53 %, 70 %, and 46 %, respectively. The performance of CNC reinforced coating was comparable to Montmorillonite nanoclay reinforced coating, while the latter providing better fruit weight retention. Pertaining to more recent studies, CNCs

have broadly been used as fillers in biopolymeric matrices for fabricating composite packaging films. Even though CNC and most of the biopolymers are hydrophilic in nature, it has proven that the resultant nanocomposite imparted increased WVBP to various biopolymer matrices (Dhar et al., 2015; George & Siddaramaiah, 2012). Formation of hydrogen bonds between the CNC and various biopolymers not only improve the cohesiveness of the film but also does not make O—H groups available for water permeation. Added to that, high crystallinity of CNC increases the thermo-mechanical properties of the nanocomposite film (Huq et al., 2012).

Homogeneous dispersion of NC into biopolymer matrix is to be ensured to enhance WVBP and OPB. One of the possible ways is to use surfactants which does not only avoid agglomeration of CNC but also act as a compatibilizer between NC and the hydrophobic matrix. Fortunati et al. compared pristine CNC-PLA with surfactant-modified CNC-PLA nanocomposites. (Fortunati, Peltzer et al., 2012) Increased dispersion of CNC and its interaction with PLA enhanced the barrier properties of the film. 1 % surfactant in the nanocomposite film formulation reduced WVP and OP by 34 % and 26 % respectively. Composites having CNC as a filler have been evidenced to have reduced water vapor permeability (WVP) and better moisture barrier properties (Paunonen, 2013). It has been testified that NC reinforced cassava starch film showed reduced WVP and increased tensile strength (Santana et al., 2019). Incorporation of NC into polysaccharides or biodegradable polymer matrices, also results in lowering down of WVP (Wang, Gardner et al., 2018).

Chi et al. (Chi & Catchmark, 2018) prepared a ternary polysaccharide polyelectrolyte complex (PPC) material via high-shear homogenization, from CMC, CS and CNC. CNC was ionically cross-linked to CMC/chitosan matrix. At 10 wt% of CNC, the Young's modulus and tensile strength were 4.7 GPa and 60.6 MPa, respectively, 60 % and 48 % higher than for the PPC film without CNC, along with a WVTR of 7982 g  $\mu\text{m}^{-2} \text{d}^{-1}$ , 40 % lesser than when no CNC was added (Table 2). The PPC material provided an impeccable barrier against grease, oil and water penetration, for up to one week, on a paperboard substrate at <5 wt% CNC concentration in agreement to previous studies (Basu, Plucinski, & Catchmark, 2017; Dai et al., 2017). The WVTR was reduced due to two reasons. Firstly, The PPC matrix (CMC/chitosan) forms a dense packed structure, refusing the water vapor diffusion. Secondly, highly crystalline CNC strengthens the interaction with the matrix (Azeredo et al., 2017; Saxena & Ragauskas, 2009). Considering a comparative study of the food packaging properties of a composite prepared from different non-wood sources of NC, Chen et al. used bamboo, sisal and cotton linters for their study (Chen, Liu, & Chen, 2019) (Table 2). The matrix chosen was starch, found to be significantly eco-friendly. (Bonilla, Atarés, Vargas, & Chiralt, 2013) It was observed

that bamboo derived NC/starch film showed the most reduced permeation towards WV and oxygen permeability, reduced by about 27.6 % and 32.9 %, respectively, when compared with the pure starch film. The reduction in the permeabilities was attributed to the higher crystallinity of the bamboo derived NC compared with sisal and cotton derived NC (Kaushik & Kaur, 2016). Table 2 displays the WVBP of NC-based composites in this section with varying concentration of constituents.

#### 4. UV-barrier and antioxidant properties

UV radiations in the range 315–400 nm (termed as UVA) and 280–315 nm (UVB) cause extensive photochemical reactions, which result in free radical formation. The free radicals further cause oxidation of lipids, proteins and vitamins, alongside, the degradation of antioxidants, change in color and texture and formation of off-flavors. Thus, exposure to UV radiation leads to loss of nutritional and organoleptic properties, reducing the shelf-life and quality of the food products (Lázaro et al., 2014; Olarte, Sanz, Federico Echávarri, & Ayala, 2009; Sadeghifar, Venditti, Jur, Gorga, & Pawlak, 2017). This highlights the importance of radical scavenging and antioxidant properties in food packaging applications (López de Dicastillo et al., 2011). PPy, a conjugated polymer, has antioxidant properties. Bideau et al. formed a composite film of NC/PPy and observed very less oxygen permeability ( $16.5 \text{ cm}^3/\text{m}^2/\text{day}$ ). At the same time, this film preserved bananas for five days without any sign of oxidation (Bideau et al., 2017). In another work, Bao et al. reported that the nanocomposite film formed with Chitosan-xylan-CNC displayed good antibacterial, antioxidant and mechanical properties wherein xylan acted as antioxidative agent (Bao et al., 2018). Similarly, Wang et al. employed epigallocatechin-3-gallate as an antioxidative agent in chitosan-BC nanocomposite and reported similar results (Wang, Xie et al., 2018). Luo et al. imitated paper-making process and fabricated aramid nanofibers (ANF)/CNF nanocomposite (Luo et al., 2019). Pure CNF displayed high optical transmittance (400–800 nm) but the UV shielding was poor as absorbance was quite low (200–400 nm). Aramid nanofibers can absorb UV rays due to conjugation effect of benzenes and amide bonds and show high transmittance in the visible spectrum. CNF film containing 2 % ANF showed excellent UV shielding with high transparency. ANFs have large number of active groups on its surface, thereby making interfacial bonding with

cellulose matrix possible. Strong CNF films can also be developed via spray coating (Shanmugam, Varanasi, Garnier, & Batchelor, 2017), or vacuum filtration (Fang et al., 2020).

Antioxidants can be combined with the food directly but are preferred to be incorporated into the packaging film. (Mastromatteo, Mastromatteo, Conte, & Del Nobile, 2010) Such films containing polyphenols like grape seed extract, murta leaf extracts, thyme extracts etc. have been employed to resist food from oxidation (Silva-Weiss, Bifani, Ihl, Sobral, & Gómez-Guillén, 2013; Talón et al., 2017). Wu et al. synthesized a multifunctional film containing TOCN immobilized silver nanoparticles (AgNPs) with grape seed extracts (GSE) (Wu, Deng, Luo, & Deng, 2019). It was observed that the film developed UV shielding, reduced transparency, antioxidant properties (due to GSE) along with increased shelf life due slower rate of release (only 5.7 % in 14 days) of antibacterial AgNPs. A biohybrid film was produced by combining anionic tannin extracts and cationic CNFs (CCNFs) (Li, Sirviö, Haapala, Khakalo, & Liimatainen, 2019). Better absorbance of UV radiations was observed by adding more tannin compounds due to the increase in polyphenolics in the film. CCNF (90 wt.%) and Tannin (10 wt.%) absorbed almost 100 % of the UV radiations (wavelength < 320 nm) whereas, CCNF (95 %) and Tannin (5 wt.%) absorbed 92 % of the radiations in the same range (Fig. 6c & d). Tannin extracts also helped reducing the haziness in the film. The antioxidant properties of the film were assessed by 2, 2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging test (Byun, Kim, & Whiteside, 2010). According to the test results, the color of the DPPH radical faded gradually due to its reaction with antioxidants. This also resulted in decreasing UV-light absorbance at 517 nm.

Cazón et al. prepared a composite by the addition of PVA and glycerol to BC films (Cazón, Velazquez, & Vázquez, 2020). The UV light (200–280 nm) transmittance of pure BC films was as low as 1 % and about 7.5 % at 400 nm (Fig. 6a, Table 2). The UV light transmittance of the BC films was further decreased to 0.57 % at 200–280 nm, on the addition of glycerol and reached a maximum value of 7.5 % at 400 nm. The transmittance of the above two films were found to be better than regenerated cellulose films and regenerated cellulose-glycerol-PVA films, respectively (Cazón, Vázquez, & Velazquez, 2018; Cazón, Vázquez, & Velazquez, 2018; Cazón, Velazquez, & Vázquez, 2019). The addition of glycerol imparted plasticizing effect (Young's modulus

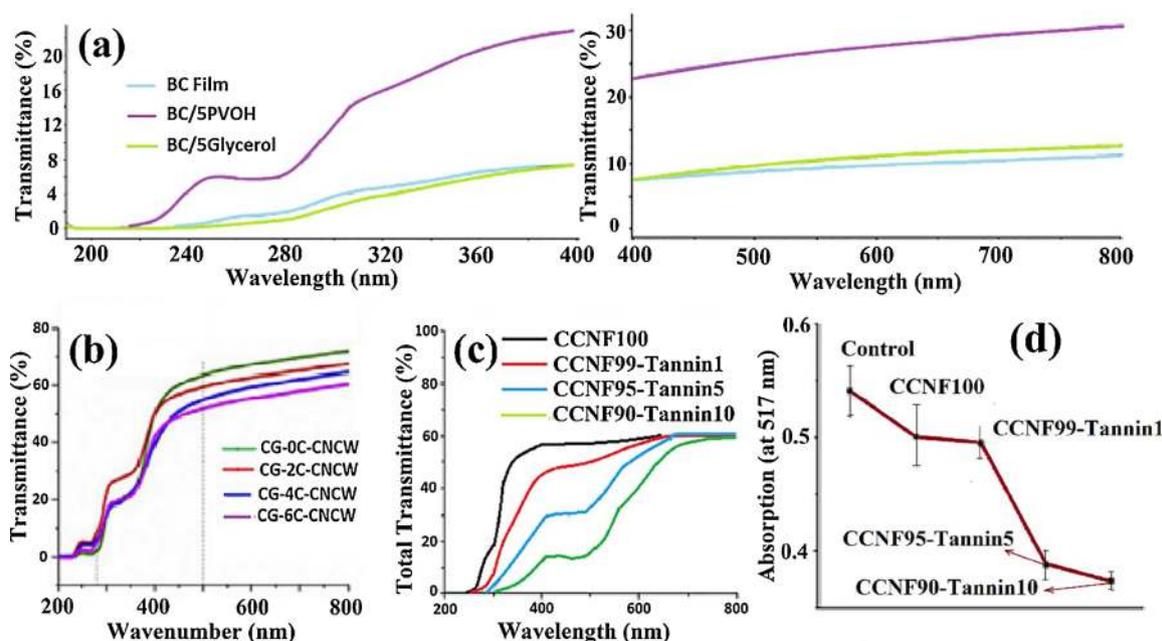


Fig. 6. (a) UV-vis Spectra of BC film, BC-5 % PVA, BC-5 % Glycerol; (Cazón et al., 2020) (b) UV-vis Spectra of CG/C-CNCW films at different C-CNCW concentrations; (Cao et al., 2020) (c),(d) UV-light transmittance and absorbance through CCNF-Tannin films. (P. Li et al., 2019).

decreased by 98.8 % at 5 wt.% glycerol) on the composite while PVA was found to improve the mechanical properties (Young's modulus improved by 17 % at 5 wt.% PVA).

Cao et al. reinforced edible packaging film with carboxylated CNC whiskers (C-CNCW), to be potentially used as oil bags (Cao et al., 2020). (Fig. 5) The UV-vis spectra of the film was studied showing that UV light (200–280 nm) barely transmitted through the film. (Fig. 6 b) The sealing ability of the film was an exceptional 1295.40 N/m for pure Cassian Gum (CG) film and 2218.78 N/m (CG-4 % C-CNCW). These values are much higher compared to a recently produced gelatin based films having a seal strength of 500–870 N/m (Ciannamea, Castillo, Barbosa, & De Angelis, 2018). It was also observed that the addition of C-CNCW to the CG films hardly affected its optical transparency. Due to low aspect ratio, CNC has less effect on transparency which is advantageous for food packaging application.

## 5. Active packaging

Antimicrobial effect, WVBP and antioxidant capacity are important characteristics of active packaging (AP) (Dainelli, Gontard, Spyropoulos, Zondervan-van den Beuken, & Tobback, 2008). Antibacterial active packaging aims at reduction or rather hindering the growth of bacteria, maintaining and increasing the shelf life of the food products (Benito-Peña et al., 2016; Lavoine, Desloges, Manship, & Bras, 2015). The introduction of antimicrobial compounds into the food packaging, directly or indirectly, can prove to be beneficial.

NC ensures high loading of the antimicrobial agents because of its high surface area. The following factors have promoted the use of cellulose in active packaging – (i) It is stable in water, thereby reduces use of organic solvents; (ii) Target surface functionalization on a vast scale favors anchoring and selective growth of nanoparticles (NPs). iii) Assist in the controlled release of bioactive compounds, into the packed food, which are encapsulated inside biopolymers. In other words, it acts as a carrier of antioxidant, antimicrobial and antifungal compounds (Fortunati, Armentano et al., 2012). Both, the compounds and NC, form essential components of active food packaging. The structure of the biofilms can be altered using CNC in order to enable better controlled release of antimicrobial agent.

The foremost advantage of active packaging is that the active biocides delivered inside the packaging, lengthens the shelf life or service

life of the food. Active packaging reduces, and thereafter, prevents the proliferation of food spoiling microorganisms (Ribeiro-Santos, Andrade, & Sanches-Silva, 2017). From the studies of kapetanakou et al. and Appendini et al. (Appendini & Hotchkiss, 2002; Kapetanakou & Skandamis, 2016), it can be inferred that there are three different ways of introducing active biocides into food packaging, as shown in Fig. 7 (Gan & Chow, 2018).

The traditional way of preventing colonization on food surface by unwanted micro-organisms is to add antimicrobial agents directly into the food formulation either by dipping or spraying techniques-(Fig. 7, concept 3). However, it causes inactivation of antimicrobials due to its rapid migration into the bulk of the food (Quintavalla & Vicini, 2002). The growth of micro-organisms occur mostly on the surface of the food. Hence, it is a good idea to release antimicrobials from the packaging film that not only controls the rate of release but also does not change the taste of food which otherwise is possible due to direct contact (Nguyen Van Long, Joly, & Dantigny, 2016). First concept can be often seen in meat packaging, whereas in concept 2, antimicrobial compounds are incorporated into the biopolymeric matrices. Bacteriocins, essential oils or polyphenols are mostly incorporated into packaging films to impart antimicrobial properties in it. Nisin is the only bacteriocin (antimicrobial peptide) which is permitted as a food preservative. In a nutshell, AP can be categorized into non-migratory (Responsive Packaging) and migratory AP (Controlled Release Packaging), categorized on the basis of release of antimicrobial agents in the packaged environment. Responsive packaging inhibits the growth of food spoiling bacteria without the actual migration of antimicrobials to the packed food (Hosseinejad, 2014). It also draws out a desirable response from the food packaging system, enabling the real-time monitoring of food quality (Brockgreitens & Abbas, 2016). Devised by LaCoste et al. (LaCoste, Schaich, Zumbrunnen, & Yam, 2005), the term CRP refers to a packaging designed specifically for the timed release of specific objects in order to maintain the safety and quality of the food for longer period of time. These two terms, CRP and Responsive AP, are supposedly the future of AP applications.

### 5.1. Controlled release packaging (CRP)

Both organic and inorganic materials can be used as antimicrobial agents in composites for food packaging. Compared to the organic

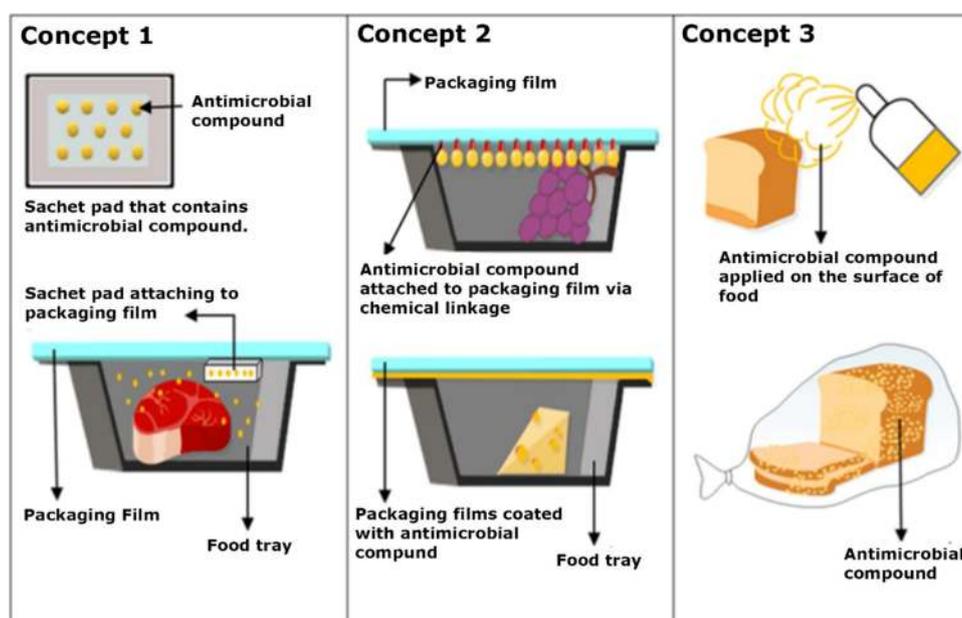


Fig. 7. Concept 1 - No contact between the food and antimicrobials; Concept 2 – Antimicrobials and food in indirect contact; Concept 3 – Both the components in direct contact.(Gan & Chow, 2018).

materials, inorganic materials such as metal oxides or metal nanoparticles, have better thermal stability; thus, can withstand more intense processing conditions. Fig. 10 graphically shows the bacterial inhibition zone thickness, produced by action of antimicrobial agents in different composite films, against different bacteria.

### 5.1.1. Inorganic particles

Some examples of inorganic antimicrobial particles include nanoparticles of Cu, Ag, Ti, Zn, etc. Among these, AgNPs are known to show higher antimicrobial behavior compared to others and provide durable and strong antimicrobial activity. It has better bactericide properties against a vast range of bacteria, viruses and fungi. (Carbone, Donia, Sabbatella, & Antiochia, 2016; Chernousova & Epple, 2013) The chemical and physical characteristics of NC are of significant importance here. The composites prepared from CNF/AgNPs have numerous applications in the food industry as components of an active packaging system. (Zhang, Yu, Wang, & Yao, 2017) The mechanism of antimicrobial action of AgNPs and the role of NC in it have been studied by a number of researchers. (Aderibigbe, 2017; Dakal, Kumar, Majumdar, & Yadav, 2016; Guo, Yuan, Lu, & Li, 2013; Prabhu & Poulouse, 2012; Su, Lin, Chen, Wu, & Wang, 2017) In a study, composite films constituting PVA, NC and AgNPs were fabricated and its effect on the thermo-mechanical properties of composite films of PVA were investigated. (Sarwar, Niazi, Jahan, Ahmad, & Hussain, 2018) Increase in tensile strength of PVA was observed and the film was successful in inhibiting bacterial activity against *Staphylococcus aureus* (*S. aureus*) and *Escherichia coli* (*E. coli*), stronger against the latter. With 16 wt.% NC at 0.3 g AgNPs, 12 wt.% NC at 0.5 g AgNPs, the inhibition zones for *S. aureus* and *E. coli* were up to  $13.6 \pm 0.68$  mm and  $14 \pm 0.70$  mm, respectively. The films formed possessed high moisture retaining capability which is suitable for storing fresh fruits and vegetables. The reason being, both PVA and NC are hydrophilic in nature, and help in moisture retention. The authors also placed their concern on the liberation of AgNPs and safety aspects to be considered before their direct use in packaging. In general, any material containing Ag, release Ag in the form of NPs or ions, prohibition of which is an important concern (Drake & Hazelwood, 2005).

The active functions of a packaging material can be enhanced using AgNPs because of their antimicrobial nature, but these nanomaterials pose toxic effects. Zhilong et al. investigated the cytotoxicity and antimicrobial effect of CNF/AgNPs nanocomposite on human colon cells (Zhilong, Wang, Kong, Lin, & Mustapha, 2019). The inhibitory effect of the composite was observed on the growth of *L. monocytogenes* and *E. Coli*, the effect being greater on the latter. There was no relevant sign of decrease in cell viability of human colon cells, tested at different concentrations of the composite (5–1000  $\mu$ g/mL), proving the composite as non-toxic for at least 24 h. Addition of CNFs in a biopolymer can display improved water and oxygen impermeability and UV barrier capacity along with an extended shelf life for fresh sirloin beef (Zhilong et al., 2017).

Functionalization of NC imparts additional properties to the resultant material. TEMPO oxidized NC bonded with carboxylate groups, which can strongly bond with AgNPs further, is one such example. AgNPs also do not allow CNFs to coalesce as carboxylate-AgNP bonding limits the hydrogen bonding of adjacent CNFs (Dong, Snyder, Tran, & Leadore, 2013). CNFs were functionalized with AgNPs by means of Tollen's reaction by Amira et al. (Errokh, Magnin, Putaux, & Boufi, 2019). Mechanical properties of the nanocomposite film containing this functionalized nanofiller with acrylic matrix was hardly affected due to the presence of AgNPs. As AgNPs were bound on the CNF surface (confirmed with TEM), the amount it leached out of the film was much below the permissible limit of 12 ppb. This composite demonstrated good antibacterial properties and proved its potential in active packaging, adhesives and coatings as well (Lokanathan, Uddin, Rojas, & Laine, 2014; Wu et al., 2014).

El-Wakil et al. successfully prepared an active nanocomposite made

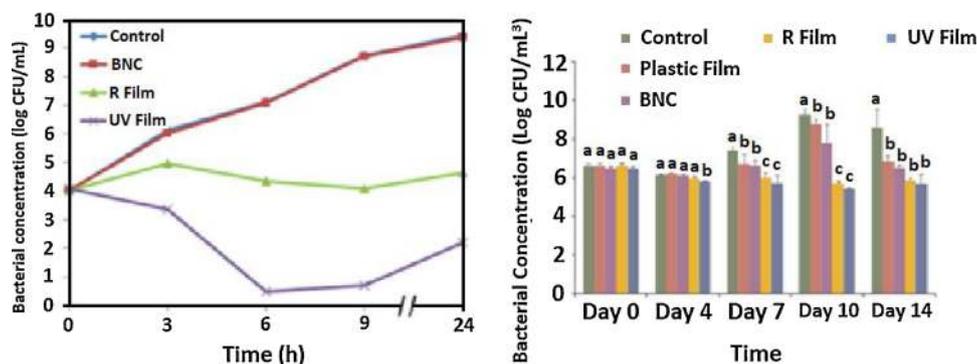
of wheat gluten, TiO<sub>2</sub> nanoparticles and CNCs that exhibited antimicrobial activity against microorganisms such as *Saccharomyces cerevisiae*, *S. aureus* and *E. coli* (El-Wakil, Hassan, Abou-Zeid, & Dufresne, 2015).

BNC is prioritized over other NCs for biomedical and packaging applications in particular because of its purity. Although, the high production cost and process optimization to widen its effective application in food packaging sector of the same has limited its research (Azeredo, Barud, Farinas, Vasconcellos, & Claro, 2019; Portela da Gama & Dourado, 2018). Films made of BNC pose great thermal stability ( $\approx 280$  °C), transparency, high tensile strength, mechanical flexibility, light weight along with commendable capacity of liquid absorption and biocompatibility (Zeng, Laromaine, Feng, Levkin, & Roig, 2014). Panaitescu et al. proposed a green solution for packaging, which was a composite produced by modifying PHB with BNC via melt processing, followed by plasma treatment or ZnO nanoparticle plasma coating (Panaitescu et al., 2018). The plasma treatment reduced the bacterial growth by 40–60%. Irrespective of the BNC concentration, it also preserved the crystallinity, melt behavior and thermal stability of the composite. On the other hand, ZnO nanoparticle plasma coating completely limited the growth of *S. aureus* along with improved mechanical, thermal behaviour and morphology of PHB-BNC, attributed to strong chemical bond between PHB and metal oxide nanoparticles.

BNC/PVA/AgNP films were produced by Wang et al. using UV-assisted method (UV) and reduction method (R) (Wang et al., 2019). Fig. 8(a) shows that, at any point of time, more number of AgNPs were released in UV film (than R film), but at a slower rate. Over a span of 24 h the bacterial concentration inside the UV film reduced dramatically, by 7.3 log CFU/mL, and by 4.8 log CFU/mL in R film. Attributed to more effective control of bacterial growth, the UV film can be useful in prolonging the shelf-life of the stored beef. They conducted another experiment to check the longevity of the effect of various films on the growth of natural bacteria on beef (See Fig. 8(b)). It was concluded that the presence of AgNPs can effectively inhibit the growth of bacteria for 10 days at 4 °C and the inhibition effect is more prominent in the beef wrapped with UV film.

### 5.1.2. Organic particles

Nisin (Salmieri et al., 2014), and some essential oils (EO) such as clove EO (Adel, Ibrahim, El-Shafei, & Al-Shemy, 2019), have been used as organic antimicrobial additives in composite films. These organic materials hinder the vital metabolism of the pathogens while some even disrupt their cell wall and functions (Dhifi, Bellili, Jazi, Bahloul, & Mnif, 2016). Each of these materials help in inhibiting the growth of specific pathogens. For instance, Lignin nanoparticle incorporated PLA-CNC film inhibits *Pseudomonas syringae* (Yang et al., 2016), nisin completely inhibits *L. monocytogenes* (Salmieri et al., 2014), etc. Plant-derived EOs on the other hand, display good antifungal, antibacterial, antioxidant and antiviral characteristics (Bakkali, Averbeck, Averbeck, & Idaomar, 2008). A composite was prepared from chitosan/ $\beta$ -cyclodextrin citrate and oxidized NC. Clove EO was added to the composite and restraint was observed against gram-negative bacteria, gram-positive bacteria, yeast and fungus (Adel et al., 2019). Abdollahi et al. developed CMC-agar nanocomposite employing solvent casting method and varied the concentration (0.5, 1, 1.5 % v/v) of summer savory essential oil (SSEO) in it (Abdollahi, Damirchi, Shafafi, Rezaei, & Ariaii, 2019). They observed that the film acted powerfully active against Gram-positive bacteria and were less powerful against Gram-negative bacteria. It was also found that the higher concentration of SSEO ( $\geq 1$  %) in the film increased water vapor permeability (due to microstructural heterogeneity) and reduced the tensile strength; both approximately by 25%. Nisin is another material having similar properties with respect to controlled release by nanocomposite material as that of EOs. Nisin (0.25–0.5 wt.%) was incorporated into CNC/corn distarch phosphate based bio-film along with  $\epsilon$ -polylysine (PL) (0.2 wt.%) by Sun et al. (Sun et al., 2019). The film showed antimicrobial activity against *S. aureus* and



**Fig. 8.** (a) Bacterial concentration with time in different films; (b) Comparison of bacterial concentration in different films, with UV films being the most effective. (Wang et al., 2019).

*E. coli*, attributed to the combination of PL and nisin. The synergistic effect of nisin and PL in expanding the inhibitory zone can be clearly seen in Fig. 9. Bagde et al. demonstrated that Bacteriocin Immobilized CNC (BIC) is a potential antimicrobial additive (57 % reduction in bacterial count) in a corn starch film (Bagde & Nadanathangam, 2019). It was observed that the incorporation of BIC also improved tensile strength (69 %) and reduced optical transparency (~28 %).

Recently, to replace AgNPs from packaging ingredients, Maliha et al. incorporated organobismuth complex (phenyl bismuth bis(diphenylphosphinato)) into NC sheets employing spray technique. The as-formed nanocomposite films were successful in inhibiting the growth of fungi and medically important multi drug resistant bacteria at higher loading of bismuth complex (>1.5 wt%) (Maliha et al., 2020) (Table 2). Zhilong et al. prepared an active biodegradable film by adding pine needle extract (PNE), lactic acid and CNF into soy protein films. Successful antimicrobial activity against foodborne pathogens (*E. coli*, *L. monocytogenes*, *S. aureus* and *Salmonella typhimurium*) along with antioxidant and light barrier properties were observed. CNF was responsible for enhancing the tensile strength and controlled release of lactic acid and PNE from the matrix that ameliorated antioxidant and antimicrobial properties (Zhilong, Dhital et al., 2019).

NC derived from natural fibers act as bioactive material as well. Fibers obtained from ginger also have bioactive chemicals in it which hinder the growth of microorganisms, showing good antimicrobial activity (Jacob, Haponiuk, Thomas, Peter, & Gopi, 2018; Zick et al., 2008). Abiral et al. investigated the antimicrobial properties of cellulose film processed from ginger nanofibers (Abiral et al., 2019). The processing involved chemical treatment along with ultrasonication. The film was able to transmit 83 % of the light at 650 nm, making it more transparent

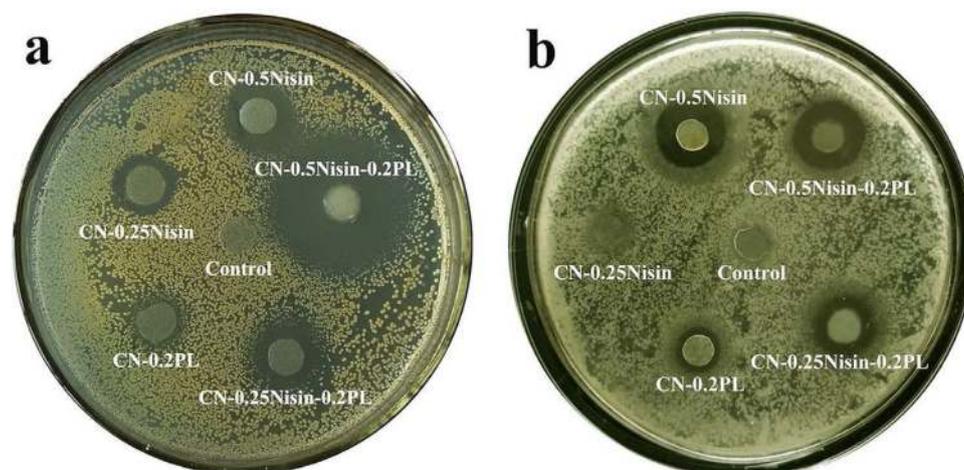
and also demonstrated good antimicrobial properties. A superabsorbent bioactive aerogel was produced using *A. donax* derived cellulosic fractions and bioactive extracts via freeze-drying method (Fontes-Candia, Erboz, Martínez-Abad, López-Rubio, & Martínez-Sanz, 2019).

The antioxidant properties of the as prepared composite were testified as absorbent pads on red meat. The aqueous extract from *A. donax* was found to have high antioxidant capacity. The color loss and lipid oxidation of the red meat was successfully reduced.

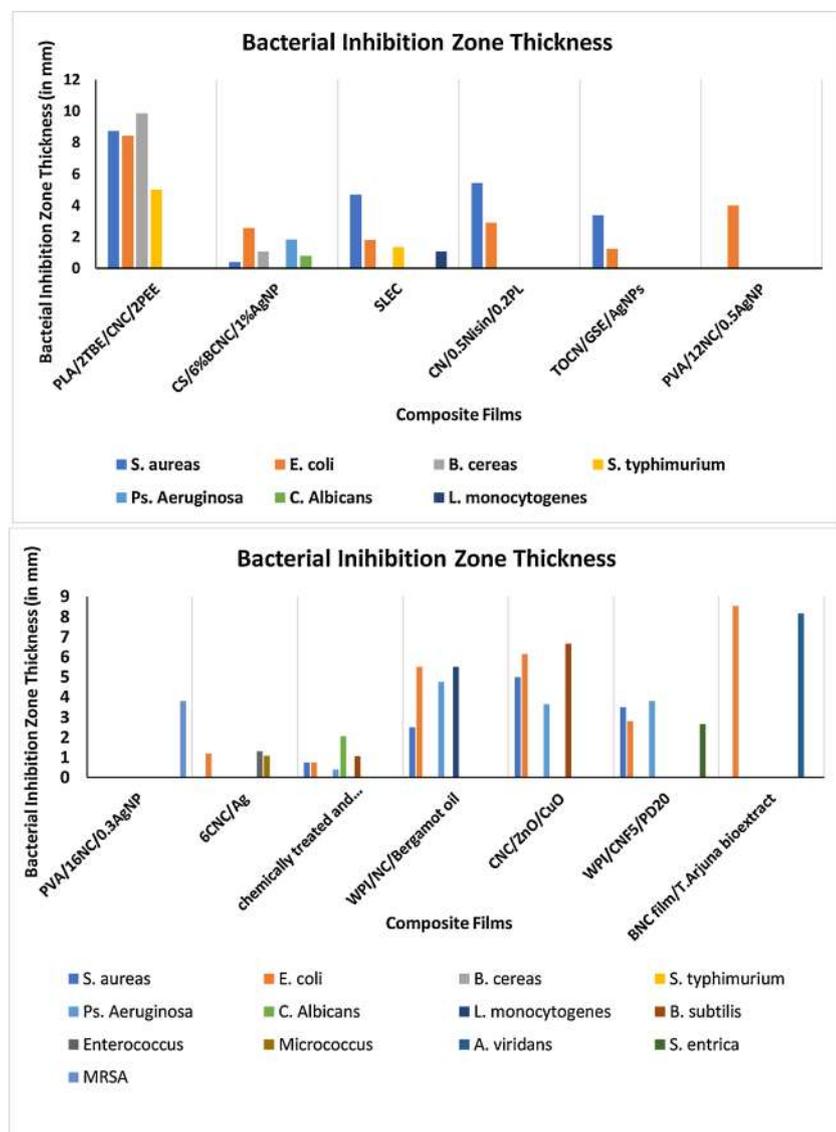
## 5.2. Responsive packaging (RP)

In simple words, responsive packaging can be termed as packaging containing an indicator which provides information on food quality and microbial activity (Lim, 2011). This can be achieved by use of different biosensors, gas indicators, time temperature indicators and barcode labels which record any internal or external changes in the surroundings of the product (de Jong et al., 2005; Poyatos-Racionero, Ros-Lis, Vivancos, & Martínez-Mañez, 2018). Responsive packaging is often termed as smart packaging, intelligent packaging or biosensing.

Recently, Vilela et al. fabricated a bottom-up built (*in-situ* polymerization) composite film composed of poly(sulfobetaine methacrylate) (PSBMA), a zwitterionic polymer showing polyelectrolyte and anti-polyelectrolyte behavior (Blackman, Gunatillake, Cass, & Locock, 2019; Yang, Zhang, Tarabara, & Bruening, 2018), within the nanofibrous structure of BNC (Vilela et al., 2019). The study was aimed at making a packaging film which could be used to monitor the humidity levels of moisture intolerant food products (meat products, dry foods or dairy products). The proton conductivity of the composite varied from  $1.5 \times 10^{-4}$  mS cm<sup>-1</sup> (at 60 % RH, 40 °C) to 1.5 mS cm<sup>-1</sup> (at 98 % RH, 94



**Fig. 9.** Inhibition zones of films incorporated with different contents of nisin and PL (Sun et al., 2019).



**Fig. 10.** Bacterial Inhibition zone thickness of different compositions – PLA/2TBE/CNC/2PEE, (Khodayari et al., 2019) CS/6 %BCNC/1 %AgNP, (Salari, Sowti Khiabani, Rezaei Mokarram, Ghanbarzadeh, & Samadi Kafil, 2018) SLEC, (Zhi-long, Dhital et al., 2019) CNC/0.5Nisin/2 PL, (Sun et al., 2019) TOCN/GSE/AgNPs, (Wu et al., 2019) PVA/12NC/0.5AgNP, (Sarwar et al., 2018) PVA/16NC/0.3AgNP, (Sarwar et al., 2018) 6CNC/Ag, (Errokhi et al., 2019) Chemically treated and ultrasonicated Ginger fibres, (Abral et al., 2019) WPI/NC/Bergamot oil, (Sogut, 2020) CNC/ZnO/CuO, (Elfeky et al., 2020) WPI/CNF5/PD20, (Karimi, Alizadeh, Almasi, & Hanifan, 2020) BNC film/T. arjuna bioextract. (C. Sharma & Bhardwaj, 2019).

‘C). The composite film prepared possessed protonic conductivity, a major asset for intelligent packaging, helping in protonic-conduction humidity sensing (Farahani, Wagiran, & Hamidon, 2014). Additionally, increased water-uptake capacity (450–559 %), high thermal stability (up to 265 °C) (placed in nitrogen environment), improved mechanical properties (Young’s Modulus  $\geq 3.1$  GPa), barrier against UV radiations were also observed. This composite film was successful in inhibiting *S. aureas* and *E. coli* bacteria as well. Sobhan et al. integrated the concept of biosensing to the smart packaging application by developing a functional NC and activated carbon (NAC) film, which combined the electrochemical properties of activated carbon (AC) with the thermo-mechanical properties of functionalized NC (Sobhan, Muthukumarappan, Cen, & Wei, 2019).

The quality of the food, which is affected by the chemical and microbial activities, can be monitored on a real-time basis with the help of freshness indicators. Visual observations (change in color of the indicators), resulting from the reactions between metabolites and indicators, form the basis for determining the freshness of the food (Biji, Ravishankar, Mohan, & Srinivasa Gopal, 2015). Changes in milk can be expressed via pH and acidity and thus the real-time monitoring of its quality is possible using suitable indicators. A smart pH monitoring label was developed by doping BNC with red cabbage anthocyanin extracts (32 and 193 mg L<sup>-1</sup>) (Pourjavaher, Almasi, Meshkini, Pirsas, & Parandi,

2017). The indicator, containing diluted anthocyanins was responsive to pH 2–10 with distinguishable color change at different pH values (see Fig. 11). The moisture absorbency of a pH indicator film directly affects the color response efficiency of the film. On addition of diluted red cabbage anthocyanin extract, nearly 15 % increase in moisture absorbency and 69 % increment on the addition of concentrated anthocyanin extract. However, the mechanical properties (Modulus and Ultimate tensile strength) of the former were better compared to the latter.

## 6. Mechanical properties

An essential requirement for good food packaging material is to have sound mechanical properties. Biopolymers have poor mechanical properties. Crosslinking or grafting of monomers has been assessed to improve the required properties (Criado, Hossain, Salmieri, & Lacroix, 2017). The presence of NC in the biopolymer increases the strength and modulus of the resultant nanocomposite. (Fortunati, Kenny, & Torre, 2019) These properties enhance with increasing concentration of NC in the matrix, up to a particular limit (approximately 4 %) (Abdallah, Mirzadeh, Tan, & Kamal, 2019; Fang, Chen, Wang, Cheng, & Ding, 2019). At this concentration, NC forms its continuous 3D network in the matrix. Above this value, the NC segregates in the matrix due to hydrogen bonding between OH groups, impairing the properties of the

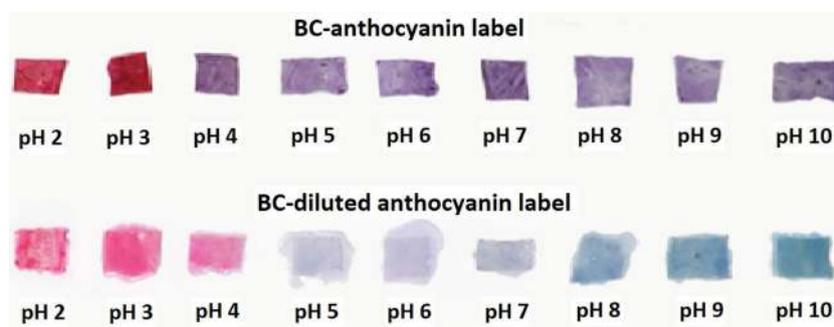


Fig. 11. Color response of BC/anthocyanin and BC/diluted-anthocyanin pH indicator at pH 2–10 (Pourjavaher et al., 2017).

composites. Segregation of NC also reduces the water vapor barrier and optical (transparency) properties. Santos et al. observed that the ultra-sonication favors NC dispersion and hence the aforementioned property enhancements (Santos et al., 2014). The biodegradable film comprising incorporation of sugar palm CNF in sugar palm starch (SPS), both considered to be agricultural residues, displayed the strength and modulus enhancement by more than 100 % at just 1 % CNF (Ilyas et al., 2019). Mechanical characterization was done on starch based composite films containing varied concentrations of bamboo derived NC, cotton linter derived NC and sisal derived NC (Chen et al., 2019). Tensile strength was greatly increased with NC addition at first but it was weakened or remained constant on further addition of NC. This was observed because large amount of NC led to its reduced interaction with polymer matrix. Elongation at break gradually decreases with increase in NC content because of rigid network formed by NC restricts the flow of starch molecular chain. B-NC was found out to impart highest reinforcing strength required for the films as it has the largest aspect ratio. In general, CNC offers better elongation properties compared to CNF and BNC as the later are subjected to entanglements due to higher aspect ratio and consequently causing agglomeration.

The mechanical properties of BNC reinforced polymer nanocomposites depend mainly on the processing method followed. Bottom-up built nanocomposites (*in-situ* process) is a process wherein, the growth of BC nanofibrils takes place (in a culture medium) in presence of second biopolymer. The same nanocomposite can also be manufactured employing impregnation of BNC into the biopolymer matrix. The earlier process has an advantage of much higher strength and modulus due to a coherent network formation and intact nanofibril arrangement (Gea, Bilotti, Reynolds, Soykeabkeaw, & Peijs, 2010). The drop in the strength and modulus in impregnation process is attributed to aggregate formation of BNC. The disadvantage with the bottom-up approach lies in not allowing to make changes in shape after fermentation.

In a case, wherein, paperboard was coated with TOCN and PPy, it

was observed that the tensile strength and young's modulus increased by 2.5 and 3 times, respectively, presence of TOCN chains were mainly responsible for enhanced strength (Bideau et al., 2018). However the elongation at break(%) decreased with a great value because of presence of PPy. Sobhan et al. prepared a film from NAC for smart packaging and observed enhanced mechanical properties (Fig. 12). High tensile strength is desired as it proves to be useful in food shipping, transportation and handling by resisting the encountered stress (Sobhan et al., 2019).

Apart from imparting antibacterial properties to the composite, George et al. observed that the AgNPs aid CNCs in increasing the tensile strength, modulus, ductility and moisture barrier of the Hydroxypropyl Methylcellulose (HPMC) matrix (George et al., 2014). FTIR study displayed the physical crosslinking due to the interactions of AgNPs with OH groups of HPMC. Sarwar et al. employed casting method to develop NC/Ag/PVA nanocomposite film and investigated the effect of NC and AgNPs on its various properties (Sarwar et al., 2018). For 8 wt% of NC into PVA, mechanical strength of the film was increased by 123 % to 12.3 MPa compared with PVA matrix. Incorporation of only 1 wt% AgNPs to this composition, not only the strength of the film enhanced by 580 % to 83.4 MPa, but also manifested non-cytotoxicity effect along with strong antimicrobial properties against both Gram-positive and Gram-negative bacteria. The increase in strength was attributed to the physical interaction between PVA and AgNPs (Table 3).

## 7. Thermal properties

The thermal stability of a film decreases with increase in the concentration of CNC. The thermal degradation of film occurs in the following steps – (i) liberation of moisture; (ii) degradation of constituents, resulting in the weight loss of the film. For instance, Cao et al. observed weight loss of the carboxylated CNC whiskers reinforced cassia gum matrix in the temperature range of 20–106 °C and 204–317 °C

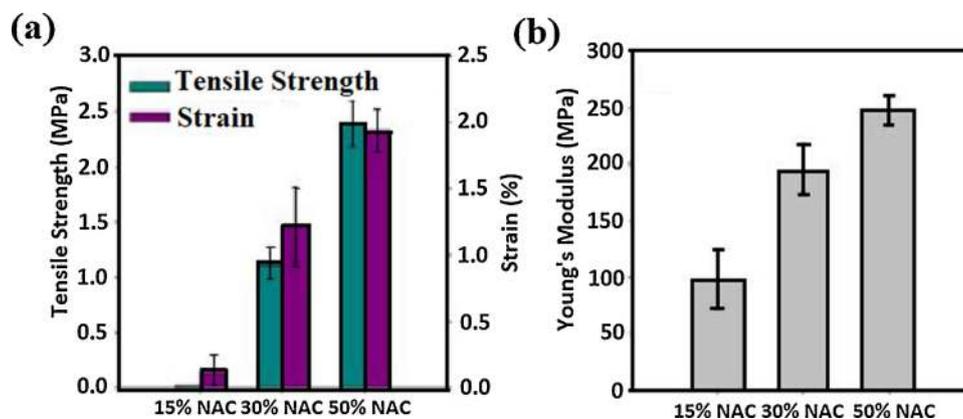


Fig. 12. Mechanical properties of 15 %, 30 % and 50 % NAC films. (A) Tensile strength (TS) and strain of NAC films; (B) Young modulus of NAC films (Sobhan et al., 2019).

**Table 3**  
Mechanical properties of recently prepared packaging films.

CNC source	Polymer	Preparation	Filler content (wt%)	$\sigma^*$ (MPa)	E* (GPa)	$\varepsilon^*$ (%)	Ref
	Matrix	Procedure					
University of Maine, USA	PBS	<i>In-situ</i> polymerization	0.5	42.1 > 54.3	0.69 > 0.83	230 > 357	(Kim et al., 2018)
MCC	PBAT	Melt mixing	7	22.0 < 12.4	0.05 > 0.08	544.5 < 567.4	(Pinheiro et al., 2017)
MCC	PBAT	Casting	10	6.3 > 7.2	0.06 > 0.12	10.2 < 5.8	(Morelli, Belgacem, Branciforti et al., 2016)
MCC	PBAT	Melt mixing	10	5.8 = 5.8	0.06 > 0.08	30 < 20	(Morelli, Belgacem, Bretas, & Bras, 2016)
MCC	PBAT	Melt mixing	0.5	11.0 < 9.7	0.04 > 0.12	927 < 558	(Zhang, Ma, & Y. Z., 2016)
MCC	PLA	Casting	5	55 < 54	1.3 > 1.6	9 < 7	(Gårdebjer et al., 2015)
MCC	PHB	Casting	5	25 < 23	1.4 > 1.8	4 < 3	(Gårdebjer et al., 2015)
MCC	PLA	Casting	2.5	40 > 51	-	4.3 > 5.6	(Espino-Pérez et al., 2013)
MCC	PLA	Melt mixing	5	43 > 46	2400 > 4400	90 < 18	(Fortunati, Armentano et al., 2012)
Cotton	PLLA	Casting	2	48.3 > 58.6	1.1 > 1.4	31.1 < 8.3	(Pei, Zhou, & Berglund, 2010)
	PHB/PCL		3	14.7 < 14.5	949 < 902	3.9 > 4.1	(Garcia-Garcia et al., 2018)
	PHB/PCL		5	14.7 < 9.3	949 > 1006	3.9 < 1.8	(Garcia-Garcia et al., 2018)
	PHB/PCL		7	14.7 < 6.5	949 > 972	3.9 < 0.4	(Garcia-Garcia et al., 2018)
	TOCN Paperboard/TOCN coated Paperboard			15.16 > 36.80	0.93 > 2.92	7.66 < 2.36	(Bideau et al., 2018)

$\sigma$  - strength; E - modulus of elasticity;  $\varepsilon$  - elongation at break. The properties were compared between CNC-based nanocomposites and neat polymer. \* > improved and < decreased properties.

respectively.(Cao et al., 2020) The factors leading to these weight losses were evaporation of water for the first, and pyrolysis of cellulose for the second.(Peng, Zhang, & Zhang, 2019) In case of films made of PHB<sub>75</sub>/PCL<sub>25</sub>, when increasing loads of CNCs were added to these films, thermal stability was decreasing. (Daniel Garcia-Garcia, Lopez-Martinez, Balart, Strömberg, & Moriana, 2018) (D. Garcia-Garcia, Rayón, Carbonell-Verdu, Lopez-Martinez, & Balart, 2017)

Panaiteescu et al. did NC and plasma treatment on PHB and found preservation of thermal stability through TGA.(Panaiteescu et al., 2018) The overall thermal stability gets influenced by plasma treatment and  $T_{max}$  was found to be increased by 1.5–4.4 °C when BNC was 1–2 wt%. However, a decrease of 5.2 % in temperature was observed at 5 wt.% of BNC, demonstrating plasma sensitivity for cellulose. (Vizireanu et al., 2018) Thermal degradation of chitin-cellulose nanocomposite was also observed by the use of TGA. The thermal stability of chitin nanofiber (CTNF) was found to be better than bamboo cellulose nanofiber (BACNF).(Hai, Choi, Zhai, Panicker, & Kim, 2020) Also with less percentage of reinforcement of BACNF thermal stability of CTNF-BACNF has very little change, although when the concentration of BACNF was increased to 25 % and 40 % then the thermal stability was found to be in between BACNF and CTNF. This gives prove that CTNF help in the thermal stability of BACNF (Hai et al., 2020).

Extraction for cellulose, can be carried via formic acid hydrolysis, TEMPO-mediated oxidation, pulp-refining and sulfuric acid hydrolysis. The CNC formed from formic acid hydrolysis has been observed to show better thermal stability and aspect ratio (Liu et al., 2016). Enzyme processed Bacterial Cellulose is more thermally stable (stable upto 378 °C) than the acid hydrolyzed one (George & Siddaramaiah, 2012).

## 8. Conclusion and inference

NC obtained from renewable resources, is biodegradable, voluminously available and sustainable, reprocessable and recyclable packaging material. However, using NC alone is not desired, as it offers poor resistance to water vapor hence is not suitable for use at high humidity levels. The presence of NC in the film as a filler or coating not only enhances the barrier properties, including gases and water vapor, but also imparts requisite mechanical properties to it. It was also observed that the elongation at break and the thermal stability of the composite

film decreased with increasing concentration of NC. The chemical surface modification of cellulose, in a way that it doesn't affect the inherent properties of same, is required.

The production of greener food packaging materials and understanding of how various antimicrobial agents behave in presence of NC when present in the packaging film has laid the foundation of new concepts in antimicrobial packaging, offering innumerable advantages in the field of AP. There is a need to identify new active agents that are responsive against a wide variety of microorganisms in AP systems. Also, focus on migration behavior of such antimicrobial agents is required as its higher amount in food is very limited as per standards (For example, upper limit for AgNPs is 0.05 mg/kg of food as per European Food Safety Authority)

In the last couple of years, researchers have attempted manufacturing of active biodegradable films employing completely green routes which does not require usage of any chemical reducing agent in the processes like plant extract reduction, UV reduction methods. BNC has interesting applications in food packaging, but not being explored much as its high production cost is a big hurdle and its commercialization for packaging market is still a challenge.

Though significant advances have been made, an equilibrium between the environmental concern, cost considerations, performance during use and the shelf-life of the film is yet far from achieving. Even though with the recent approaches of making controlled release of antimicrobial agents in presence of NC possible; using bio-polymers as raw materials having desired barrier properties, tensile strength and meeting the food packaging requirements is still a big challenge to overcome. To exploit NC in commercial packaging, material scientists, and engineers should work hand-in-hand. The prevalent employment of such a biodegradable and sustainable NC replacing its plastic counterpart will surely contribute in reduction of the greenhouse gases.

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