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REVIEW ARTICLE

Paper and other pulp based eco-friendly moulded materials for food packaging applications: a review

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ABSTRACT

Pulp is one of cheap resources with abundant availability. Papers are made using pulp which finds tremendous use in packing the food products. Such materials are also can be recycled and reused to develop moulded articles useful for packaging applications and other food contact items like trays, cup, plate and pouches etc. Last decade has evidenced several advancements in manufacturing technologies and recycle technologies for paper and pulp based moulded articles. However, there are also many constrains and challenges that were faced during manufacturing of products. This review article covers the various papers and pulp based moulded articles that can be used as food contact materials along with their manufacturing and recycling technologies. Moreover, this article also briefs about various characterization techniques used to ensure the quality of paper and pulp based moulded articles. This information is beneficial in making progress in the development of moulded articles using paper and pulp.

Keywords: Paper, pulp, moulded article, waste, manufacturing, recycling, characterization techniques

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INTRODUCTION

Paper as well as other pulp based products are considered as a replacement of single use plastic in food packaging applications due to its eco-friendly nature, abundant resources, low price, low weight, good mechanical property, biocompatibility and recyclability (Youssef and El-Sayed 2018). Papers are made from the pulp materials. The raw pulp contains ~96% water and requires multi-step processing to obtain paper and other moulded items (Deshwal et al., 2019). Newspaper (Another material made of pulp) has become a popular choice for providing cushonic effect as well as for providing protection to food items. Various types of papers are in use in food packaging in the form of paper bag, composite cans, fibre drums, multiwall paper sacks, rigid boxes, folding cartons, corrugated fibreboard, paper bottles (e.g. pure-Pak, Tetra Pak for packaging liquid materials like milk, juices etc.). Advances of research regarding the transformation process of the pulp and paper to the usable form for packaging products is one of the key interests of research today. Moreover, bio-composites are fabricated by blending various natural materials like sugar beet pulp, mulberry pulp, microfibrillated celluloses etc. with synthetic polymer like PVA, chitosan, alginate for the use in packaging applications (Youssef and El-Sayed 2018; Shen et al., 2015; Wang et al., 2017). International Mold Fiber Association (IMFA) provided guidelines for moulding fibres originated from natural resources to make it suitable for food packaging applications. These fibres are a rich source in renewable packaging

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materials. IMFA highlights many applications of such materials like "clam-shell, carryout food containers, cups, bowls, plates, food and serving trays, egg, trays and cartons, punnets, lamination, locator trays for bottled products, wine, jellies etc." (IMFA 2020). Application of such moulded paper and pulp based products in various packaging is defined by its grades and quality. Many types of papers and paper board are available which are made from either pulp or recycled paper waste using different manufacturing procedures. The characteristics of moulded articles depend on the resources used, manufacturing procedure as well as recycling technologies. These are the topic under focus in this article. The types of paper, paper board and other moulded fiber products are discussed in the subsequent sections. However, such moulded articles suffer from serious drawbacks like (i) moisture sorption which directly affect the static compression strength (Sørensen and Hoffmann 2003), water vapor transmission (Sørensen and Risbo 2005), oxygen permeability (Jabeen et al., 2015), brittleness, thermal stability, poor impact strength (Jabeen et al., 2015) which limits its use as packaging materials. But major drawback is its limited resources. Jabeen and Nayik (2015) reported many uses for cellulosic materials which are listed in Table 1.

Table 1: Nature derived biopolymers used in developing packages

Materials	Main Materials	Possible sources	Company	Packaging Applications	References
Bio-based trays wrapped with cellulose film	Cellulose	Plant	Wal-Mart	Kiwi	Jabeen et al., 2015
Metalized cellulose film	Cellulose and metallic layer (Mainly Aluminium)	Plant	Boulder Canyon, Qualitystreet, Thomton	Potato chips, sweets	Jabeen et al., 2015
Cellulose based packaging	Cellulose	Plant	Birkel	Organic pasta	Jabeen et al., 2015
Natural continuous bast fibers	Cellulose, lignin	Hemp, flax	-	Composites, replacement of glass fibers	Lebrun et al., 2013
Binder free cellulose fibers	Cellulose, lignin etc.	flax fibres, cotton stalks, hardwood and softwood	-	cellulosic panel	Arevalo et al., 2019
		pulps, softwood sawdust, kraft lignin and banana bunch			
Wheat Straw (Triticum Aestivum PBW-343)	Holoc, alphacellulose, hemicellulose, lignin	Cellulosic fibres	Multiple companies	Paper and paperboard	Singh et al., 2011
Waste papers	Cellulose	Recycled papers, cardboards, newspapers, magazines, phone directories	Multiple companies	Egg trays, food trays, cup, plate etc.	Martínez et al., 2016

However, last decade has evidenced several advancements in processing technology for paper and pulp based moulded articles as well as development of new products. Hot pressed cellulosic composite is one example of such development which is found to have excellent flexural strength (75 MPa) and a modulus of 5.9 GPa (Arevalo et al., 2019). Cellulose based nanofiber board is reported which possesses maximum flexural strength of 162 MPa and tensile strengths of 85 MPa (Yousefi et al., 2018). Nano-fibrillated cellulose gains popularity in recent days due to having characteristic like self-binding ability, high modulus and low cost. This review article briefs about the classification of paper and pulp based products and its various manufacturing technologies, recycling technologies and characterization techniques; these are required to develop moulded articles with required set of properties and to maintain the quality of the products.

CLASSIFICATIONS

Moulded pulp products are classified into four categories based on the production process, fabrication techniques and the density of the products (Didone et al., 2017); these are: (i) Thick wall, (ii) Transfer moulded, (iii) thermoformed or thin walled and (iv) processed. Details specific to all the categories of the molded pulp products are discussed in Table 2.

Table 2: Characteristics, requirements, raw materials used and application of various types of moulded pulp products

Category	**Mould requirement**	**Raw material**	**Product characteristics**	**Application**
Thick wall	Open mould with a smooth inner surface and rough outer surface	A blend of kraft paper and recycled paper	Thick walled product with 5-10 mm thickness	Support packaging for non-fragile and heavy items
Transfer Mould	A forming mould and a transfer or take-off mould	Recycled newspaper	Relatively thin walled (3-5mm thickness) with relatively smooth surfaces on both sides and better dimensional accuracy	Egg trays and packaging for electronic equipment
Thermo-formed (thin wall)	Hot press mould	Partially formed products using paper and pulp.	high-quality, thin-walled products with thickness ranges from 2 to 4 mm and with good dimensional accuracy and smooth rid surfaces	Various thermoformed items.
Processed	Not Applicable	Paper and pulp products that require special treatment	Printed /coated surface	printing, coatings, or additives

On the other hand, paper made from the pulp is classified into mainly four categories. These are Parchment/Baking Paper, Greaseproof Paper, Kraft Paper and Corrugated board. Acid treated pulp is used to make parchment paper which possesses better oil and water resistance. This paper finds its use in packaging butter and lard. Grease proof paper has better oil resistance and generally is used to wrap various oily foods like snack foods, cookies etc. Kraft paper is again classified into four categories which are natural brown, unbleached, heavy-duty and bleached white. Kraft paper more specifically natural kraft finds enormous use as bag for packing fruits and vegetables. Corrugating flute paper (CFP) is laminated with two layers of kraft paper (one is placed above CFP and another is place beneath it) to obtain corrugated board with high impact resistance property. It is very useful for packing bulk foods for export purposes. Paper board is available in many grades which are solid bleached board (SBB), solid unbleached board (SUB), folding boxboard (FBB) and white lined chipboard (WLC) (Riley et al., 2012). Configuration of these grades is shown in Figure 1.

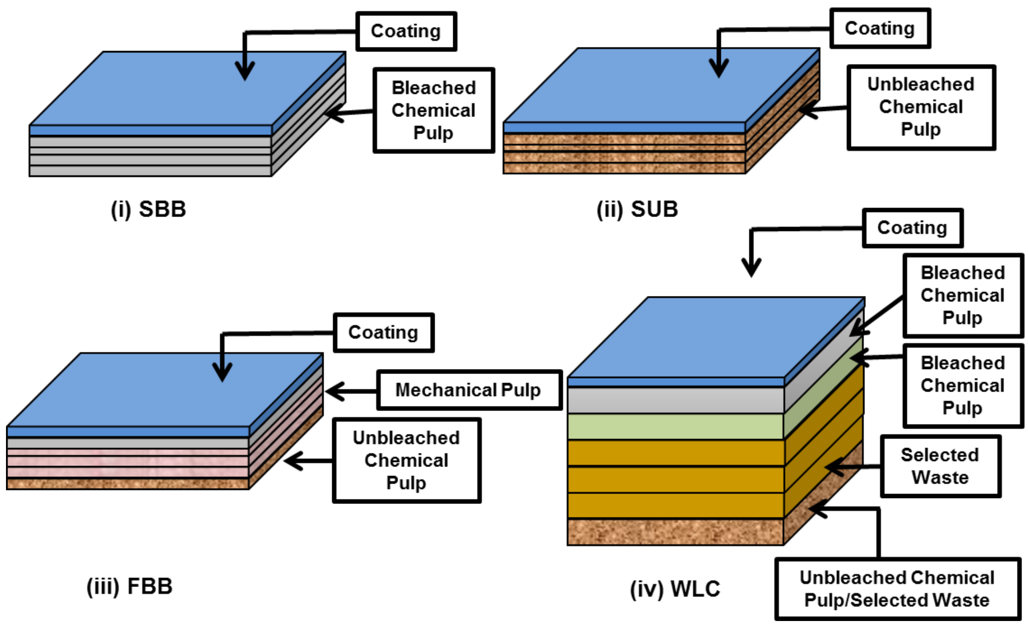

Figure 1: Configuration of different grades of paperboard (Riley et al., 2012)

There are two other popular grades used extensively in the market for its excellent durability in packaging applications. These are Clay Coated News Back (CCNB) and Clay Coated Kraft Back (CCKB). In case of CCNB, the pulp layer which is beneath the coating layer is made of newspaper whereas, in case of CCKB the layer is made of "virgin sulphate unbleached pulp or fibers" recovered from used corrugated boxes (Riley et al., 2012). According to Riley et al. (2012), DN19303 defines various grades of paperboard which are used by United States, United Kingdom and European cartoon manufacturer (Riley et al., 2012).

PRODUCTION PROCESS

Martin L. Keyes registered the first patent for a machine for making pulp products in 1903. Canadian inventor Joseph Coyle developed a procedure to use moulded pulp in packing eggs (Didone et al., 2017). After the First World War, the advancement in the development of production machineries was initiated. Patent registered in the year 1920 on the multi-use packages is the initiation of new era in pulp or paper based packaging technology (Didoneet al., 2017). Since then, many development of different forms of packages were evidenced. Few examples are folding spoons, pastry holder, packaging for handset, fruit trays, drink carriers, packaging for various food items, moulded honeycomb paperboard etc. (Didone et al., 2017). In addition, last decade evidenced many developments in technologies for making various packaging solutions like laundry-detergent bottle, Pangea Organics packages, Paper Water Bottle, green fiber bottle which is made of pulp (Didone et al., 2017). It is noteworthy to mention here about the process for developing Tetrapak which involves two manufacturing steps. Didone et al describes the steps as "vacuum forming of the pulp into the desired shape and drying the product to remove the remains of the water" (Didone et al., 2017). During the last few years, interest was found to grow enormously for developing effective tool for moulding the products. Numerous types of tools were developed for moulding the pulp to obtain desired packaging solutions to fulfill the purpose like (i) replication of the desired geometry, (ii) maintaining the surface finish quality, (iii) easy processing and cleaning between production cycles, (iv) excellent corrosion resistance, (v) no adherence characteristics for the pulp materials and (vi) long service life i.e. the tool should sustain a large number of stress cycles.

Transformation of pulp to moulded articles is composed of several steps. In the first step, paper and vegetative residues are mixed with hot water and kept in this condition for 20 minutes to convert it into pulp materials. Purification and separation procedure are required for the materials like cardboard, newspaper, magazines, recycled products etc. for obtaining the pulp materials. Then the mould is immersed into the stock pond (tank filled with pulp). Here, the pulp material is sucked onto the mould by vacuum assisted process from the stock pond. The bound water is removed using vacuum. In the subsequent process, wet pressing method is used using one rough surface and one smooth surface. Here, the wet fibers are pressed between two platesto expel out the water from the materials. Platen press arrangement is found to be beneficial for pulp making process. In this process, water flows into the fiber network where a viscous force is experience by the water. The interaction between fiber and water results in a non-uniform compaction of the wet web in the longitudinal direction which is known as stratification. In the next process, the product obtained is subjected to drying process where the oven drying is mostly preferred. Compression moulding, thermoforming are the next advancement which is used to produce the thin walled product in the desired shape and size with high degree of accuracy. Søllner (2016) suggested a method for producing moulded products (Søllner, 2016). Here, the product is transferred from forming tool to drying tool via a rotating table where the article is pressed from inside and bound water is extracted through a porous mould by vacuum. The process provides many benefits like low energy consumption, low cycle time, low production cost etc.

Lebrun et al. (2013) elaborated the process for making the sheet of paper from pulp (Lebrun et al., 2013). The development procedure for sheets of paper includes (i) dynamic sheet former, (ii) sheet press and (iii) sheet dryer (Lebrun et al., 2013). The dynamic sheet former is composed of mainly three parts which are projection nozzle, spinning drum and a container with stirrer. Here the pulp (dispersed in water) is sprayed inside the rotating drum to form a thin sheet of wet layers over the porous inner surfaces. Due to centrifugal force of action, the bound water exudes out from the wet pulp layer. In the subsequent process the wet layer is pressed under a series of increasing pressure condition to obtain a sheet using a sheet press. The sheet press consists of a pressure roller and an absorbing sheet felt. Here, Lebrun et al. (2013) reported the use of natural bust fibers (Unidirectional flax fiber sheet) as an intermediate layer which was placed between two papers of 75 g/m2 and pressed to obtain a reinforced sheet to achieve higher mechanical properties. A small amount of water is suggested here to spray over both the flux and the paper surfaces before introducing it into pressure roll. Then the sheets are dried using a sheet dryer to obtain the final product (Lebrun et al., 2013). Moreover, the sheets can also be dried in oven where it is subjected to heating at temperatures ranges from 140 to 240 ˚C for 10-15 minutes (Martínez et al., 2016). The egg and food trays are made at this stage. However, other products like cups, plates etc. require further pressing and printing (Martínez et al., 2016).

Manufacturing technology of pulp molding plastic product from waste paper was well described by Li and Xiao (Li and Xiao2013). Waste paper is a good selection as a raw material as it has abundant resources and low cost. It is necessary to classify the waste paper before processing. The classification can be made on the basis of sources, collecting funnel, quality and wastepaper fiber types. Generally, the post-classification processing procedure has four steps which are: separation, optimization, purification, drinking, concentration, bleaching and pulping. There are five types of procedure for moulding the pulp. They are described in Table 3.

Table 3: Description of different types of procedure of moulding pulp materials (Li and Xiao 2013)

Types	Description	Type of environment	Principle
Vacuum fine pulp molding process	A mould with wire net into the attenuate pulp	Vacuum	Vacuum assisted extraction of water from the pulp through wirenet
Casting method	A mould of wire net joined with a pulp box through a flow channel	Flow pressure	Pulp is allowed to flow from a pulp box to a mould of wireframe. The water is expeeled out through the mesh.
The method of hydroform	Batcher, pump, mould, reticular membrane	Hydraulic pressure to compact the fiber, vacuum to suck the mould and compressed air to separate the product	The pulp materials are first extruded through a extrusion head over a recticular membrane. Then hydraulic pressure is used to compact the materials to exude out the water from the pulp. The vaccum is used to suck the product over the extrusion head and then compressed air is used to separate the product from the extrusion head
Compressing air method	Pulp vat, stuff pump, batcher.	Compressed air	Compressed air is used to press the wet pulp material to exude out the water from it through the wirenet/support membrane.
Compact setting wringing	Compact setting equipment	Hydraulic pressure or compressed air	It is a setting and wringing purpose by compacting the pulp between two membrane

Two major types of process are used for pulping the waste cellulosic materials like paper, cardboard etc. and the other allied natural fiber based materials. These are (i) mechanical process and (ii) chemical process. Chemically processed pulps are of higher cost compared to the mechanically processed pulps. However, the mechanically processed pulps require less time for production compared to the chemically processed pulps but quality of the paper produced from the mechanically processed pulp is relatively poor. Papers from mechanically processed pulp are generally used in newsprint papers or as a blend as well as intermediate layer for chemically process pulp based product. This type of papers is only used with the aim of reducing cost of the materials. This is also the quickest and economic method of obtaining virgin fibres.

Mechanically processed pulps are generally produced by cleaning-grinding-compaction process (Riley et al., 2012). Here, the wood chips are thoroughly washed to remove any contaminants like dirt, soil, stones etc. Then the material is subjected to the grinding process using grindstones or rigid metal discs to grind the logs directly. The process separates the fibers but results in a reduction in fiber length which is the main disadvantage of this process (Riley et al., 2012). Here, the lignin is not separated from the product. But in the chemical process, the lignin is separated from the cellulosic fibers by the heat and chemical treatment. The chemical process is of two types which are alkaline sulphate process (Kraft process) and acid sulphite process.

Chemical digestion is used in case of both the procedure to obtain white fibers (free of lignin). Sodium hydroxide and sodium sulphite are used in case of Kraft process for chemical digestion whereas metal or ammonium salts of sulphurous acid (This chemical produces either sulphites or bisulphites) is used in the sulphite method. The product obtained has higher strength and high degree of purity (Riley et al., 2012). The sulphite process is used for producing only 20% of the pulp which find its use in developing products like rayon, cellulose acetate and cellophane. These products are suitable for packaging of fresh food items, twist wrap films etc. Characteristics like breathability and excellent mechanical strength as well as the excellent dead fold characteristic make it a popular choice as material for packaging applications (Riley et al., 2012). Thermo-mechanical (TMP) and chemical thermo-mechanical (CTMP) are the two intermediate processes which gain popularity in the pulping process technology. Hot water treatment is used in case of TMP process which results in softening of the fibers during mechanical process to reduce the possibility of damaging fibers. The CTMP process is a combination of heat and chemical treatment which is found to be more effective in reducing production cost. This process is mainly used in making corrugated fluting medium, liquid packaging containers (Riley et al., 2012).

Thumm and Disckson (2013) reported the use of pine pulp for the reinforcement of polypropyne composites (Thumm and Dickson2013). A 2-stage radiata pine slabwood TMP fiber of fiber length lies in the range between 3-25 mm is used here. Beside this, many biopolymers derived from natures find use in packaging applications. Tumwesigye et al. (2016) describes an integrated sustainable process for developing packages using cassava starch (Tumwesigye et al., 2016). Cassava is available in two forms: sweet and bitter among them sweet cassava is consumable. Last decade has evidenced a paradigm shift towards a fully industrial-applied sweet cassava as ~162.5 million ton is produced only in Africa (Almost half of total production of Cassava in the whole world) (Tumwesigye et al., 2016). The steps followed by industries to extract cassava starch from non-edible parts and cassava chips are: (i) preparation (peeling and washing), (ii) rasping/pulping, grating, (iii) recovery (starch sedimentation, washing, dewatering, drying) and (iv) finishing (milling and packaging) (Tumwesigye et al., 2016). Preheating-pasteurization-functionalization-casting process is adapted for the development of packages as reported in many literatures. Beside this another process is described by Curling et al. (2017) to make packaging materials using wheat straws (Curling et al., 2017). Here, the straws are first fed into a cooker or digester where steam pressure is maintained at 0.94 MPa using steam at 390 ˚C. Then the straw is fed through a refiner; here it is passed in between two refiner plates of 30 cm in diameter and of parallel bar configuration.

Moreover, some essential oils (EOs) like Carvacrol, oregano, and cinnamon EOs are reported to be used along with βCD (Kleptose®10) and Waterproof lacquer (UKAPHOB HR 530 having 10.5% solid content) as an active coating materials which is found to enhance the storage life of fresh Mandarins significantly. Kneading method is used to prepare EOs−βCD inclusion complex where the components are taken in 1:1 molar proportion (López-Gómez et al., 2020).

RECYCLE OF PAPER BASED PRODUCTS

Paper or paper based products can be easily recycled through cleaning, and washing, re-pulping and de-inking, pressing and drying, printing, storage (Wistara and Young 1999). It can be reshaped to new articles for using in many packaging applications. However, such recycled materials as well as recycle technology have many issues and last decade has evidenced several technological advancement on addressing these issues which are noteworthy to mention here. In most of the processes, the material has to undergo through several physical and chemical changes where a reduction in strength of the materials is obvious. The drying process during recycling results in partially irrecoverable closure of pore wall and thus causing for increase in resistance for wetting and partially irreversible loss of flexibility. This phenomenon is known as

Hornification. Such phenomenon benefits the material during application but at the same time limits its recycling. The recycled paper is found to exhibit lower mechanical properties than the virgin pulp based products. Intrinsic fiber strength, inter-fiber bonding and inter-fiber contacts (Mao et al., 2017a; Mao et al., 2017b; Goutianos et al., 2018) are there essential parameters which are found to be affected during recycling. During processing, many hydrogen bonds are formed among cellulose fibers which are not affected during recycle process. Such existence of hydrogen bonds caused hornification which reduces the swelling attributes of the paper based materials. Hence, it reduces the inter fiber contact as well as inter-fiber bonding which inturn results in a reduction of intrinsic fiber strength. This is one of the major limitations for recycling waste papers. Moreover, printed surface and growth of microorganisms pose challenges to maintain product quality developed using recycled papers. The Deinking process (This is used for printed materials) is of three types; these are (i) physical method, (ii) chemical method and (iii) enzymatic method (Saxena and Singh Chauhan 2016). Ultrasonication, microwaving and high temperature are used as physical method for deinking process. Ultrasonication is an effective process which separates the ink residue adhered over cellulose fibers and the separated particles are then fragmented to smaller particles by ultrasound. Zhenying et al. (2009) integrated the ultrasound process with UV irradiation and enzymatic methods (Zhenying et al., 2009). Laser printing toner is composed of thermosetting resin which crosslinks after application. Such crosslinked solid particles are difficult to separate even through ultrasonication. Ultraviolet assisted degradation of the thermosets is evidenced as effective strategy for removing the thermoset inks from the surface. A combination of physical methods (Sonication and microwave treatment) with enzymes is another effective process for deinking newspaper. The enzyme S-MW-XL is used in this case for deinking process (Saxena and Singh Chauhan 2016).Chemical deinking process is the treatment with alkaline agent like sodium hydroxide, bleaching agent like hydrogen peroxide, chelating agent like diethylenetriamine pentaacetic acid (DTPA), ethylenediamine tetraacetic acid (EDTA) etc., sodium silicate (chelating and buffering agent), surfactants and other chemicals. However, the use of physical process and enzymatic process are evidenced to reduce the consumption of alkali in the waste paper recycling. As alkali is more hazardous and at the same time is a pollutant that contaminate environment, both the physical process and enzyme assisted deinking process make the recycle of waste papers more eco-friendly (Saxena and Singh Chauhan 2016).Tsatsis et al., reported enzymatic treatment to facilitate the deinking from printed paper surfaces. Novozymes 342, Cellusoft CR, Accellerase 1500, Cellusoft L are the few examples of enzymes which are used for commercial cellulose preparation during enzyme assisted deinking process reported by Tsatsis et al. (Tsatsis et al., 2017). Optimum temperature and pH range for this process depend on the enzyme used. Saxena and Chauhan described in their review article about the role of various enzymes on the deinking process (Saxena and Singh Chauhan 2016).The commercial cellulase enzyme "OPTIMASETM: is compared with the chemical deinking process report of which shows a 24.6% improvement in ink removal efficiency along with 15.3 and 2.7 % increase in burst strength and tear index respectively (Pathaket al., 2011). Low chemical requirement and lower residual ink are two main advantages of enzymatic process over conventional chemical treatment (Saxena and Singh Chauhan 2016).

Microorganisms developed on waste papers are another problem which evolves some issues like odour, discoloration etc. This is manly associated with the discarded paper which is used as food contact materials. Such microbial problems are documented for decades. Antimicrobial packaging is endorsed to mitigate such problem and endorses the prevention of growth in microorganisms. A paper mill can fulfill every requirement which promote the growth of microorganisms. The pathogens can enter to paper at any stages and water, additives act as carrier for such pathogens. Being biodegradable, the lingo-cellulosic materials becomes the perfect medium for the growth of microorganisms. Water and the temperature ranges between 30-45 ˚C in paper mill fulfill the other demand for the growth in mesotrophic organisms (Flemming et al., 2013). The microorganisms converts the lingo-cellulosic materials to metabolic products and forms biomasses, folks etc. The problems due to growth of microorganisms encountered are listed below (Flemming et al., 2013):

1. Sticky matrix
2. Discoloration and loss of stability of the pulp
3. Malodor formation
4. Accumulation of particles resulting in “stalactites of slime”
5. Unwanted dislocation after reaching a critical mass of slime aggregates
6. Holes on the paper surface which results in deterioration of mechanical properties

Chlorine is used as bleaching agent for decades which has well known antimicrobial characteristic and is evidenced to suppress the growth of bacteria effectively. But, the use of such chemical is banned due to its hazardous effect on human health as it produces toxic bi-products like chloramines and trihalomethanes (Watson et al., 2012). Now-a-days it is switched to peroxide bleaching but it has poor antimicrobial activity. This is evidenced to increase in formation of “pink slime” which further leads to “salmon-red deposits”. This results in discoloration of the paper (Salzburger 1994; Flemming et al., 2013).

CHARACTERIZATION TECHNIQUES

For the paper and pulp products, pH, drainage and distribution of particle size are measured prior to the molding process. The drainage is characterized using the standard method ISO 5267-2 (Curling et al., 2017). Usually, particle size is characterized by drying the pulp to obtain the dry fibers and then sieved through a sequential series of wire mesh based on the mass fraction. Paper and pulp products are also chemically characterized to obtain information about the presence of wax, klason lignin, alpha cellulose and hemicellulose, ash content etc. in paper and pulp products. Percentage of wax present in the sample is estimated by dry weight of the remaining residue after soxhelet extraction (at least 50 solvent cycles) using a solvent mixture of toluene, acetone, and methanol (4:1:1) for 8 h using soxhlet apparatus. Klason lignin is determined as a percentage on dry weight basis following the procedure prescribed in TappiT 222 (Tappi 2002). 72% sulphuric acid is used here to hydrolyze the polysaccharides present in the sample and after the process of hydrolysis lignin remains as solid residues. Sodium chlorite method is used to determine the alpha cellulose and hemicellulose content. Here, sodium chlorite is used for delignification of the sample. In this process, lignin is oxidized to obtain acid which is removed from the samples through extraction procedure and the remaining residue contains cellulosic particles or fibers. This cellulosic residue is then extracted using 17% sodium hydroxide solution and neutralized using acetic acid. The dry weight of the residue left is used to evaluate the content of cellulose present in it. For evaluating the hemicellulose content, the ethanol is added to the filtrate which results in precipitation of hemicellulose material. Residue is then separated from the aliquot by either filtration or centrifugation method and dried. The weight percentage of this dry weight of the residue indicates the percentage of hemicellulose in the sample (Curling et al., 2017). Characterization of the moulded articles is equally essential to maintain the quality of the food packages. Majorly the characterization techniques used for these types of materials, are briefed in the subsequent sections.

Thickness measurements (Micrometer)

Thickness plays a crucial role in determining the mechanical durability for paper and pulp based products. Generally, micrometer is used to measure thickness. Thickness is determined for paper and paperboard product using the method described in ISO 534-2005(Adamopoulos et al., 2014). Adamopoulos et al. (2014) carried out thickness test of grade paper made from recycled pulp using the guidelines given in ISO 534-2005.Tappi T 411 (Tappi 1997), contains the guidelines of testing the thickness of single sheet and the variations in single sheet paper, paperboard and combined board using automatically operated micrometer, when a specified static load is applied on it.

Permeability measurement

Permeability is found to greatly impact the mechanical durability of paper and pulp based products as all of them are hydrophilic in nature. A resin infusion setup can be used to determine permeability which has two parts: (i) a flat plate and (ii) a vacuum bag mounted with a central injection point. In this case, a flexible diaphragm is used instead of rigid mould (Lebrun et al., 2013). The experimental set up for this type of permeability measurement is described by Lebrun et al. (Lebrun et al., 2013). The equivalent permeability for this procedure can be estimated for anisotropic reinforcement using the following equation:

$$F = \left(\frac{R_{x,e}}{R_{x0,e}}\right)^2 \left[2ln\left(\frac{R_{x,e}}{R_{x0,e}}\right) - 1\right] = \frac{4K_e.\Delta P.t}{\varphi\mu R_{x0,e}{}^2}$$

Here, $R_{x,e}$, $R_{x0,e}$ are the equivalent radius and the equivalent inlet radius respectively. ΔP is the pressure gradient between flow front and the gate. The parameters t, φ, μ are the elapsed time, the reinforcement porosity and the resin viscosity respectively. F is the dimensional factor which is plotted against t and from the slope of the curve, the equivalent permeability (K_e) can be determined.

Water absorption

Cobb test is performed to determine the water absorption for the materials which exhibits complete absorption of water in more than one minutes as per the guidelines in ISO 535-2014for the paper or cardboard type of materials (ISO 2014). However, for porous paper such as blotting papers, newspapers with higher water absorption another method is available which is ISO 8787:1986 (Klemm method) (ISO 1986). Klemm method, the sample specimens are dipped to a set depth in water and after a set time the change in height of the water level is measured (Riley2012). Tappi 432 is available for testing water absorption for bibulous paper (Riley2012). Cobb test for measuring water absorption of non-bibulous paper, paperboard and corrugated fiber board (CFB) is elaborately given in TAPPI 441 om-09 (TAPPI2009). In this metho,d a metal cylinder having cross sectional area 100 cm^2 is placed on top of the sample cut in dimension (10cm*10cm), and is clamped tightly. 100 ml water is poured into it till 1 cm height for a time period of 1 minute (for paper) and 30 minutes (for paper board and CFBs). At 10±2 s before completion of the predetermined test time, the water is carefully poured out and the sample is weighed after removing excess water from the contact surface. This final weight obtained is used to find to water absorption using the formula:

Water, g/m^2 = (Final weight, g - Initial weight, g)*100

The obtained value indicates the water absorption capacity of the packaging material, which is a very important criterion for determining the stability of the packaging. ISO 535:2014 (E) also provide the guidelines for measuring water absorptiveness by Cobb method. The method of water absorption test is also mentioned in IS 1060 (Part 1)-1966 (IS 1966).

Moisture content

ISO 287-2017 (ISO 2017), IS 1060 (part 1)-1966 (IS 1966) and TAPPI T412 (TAPPI 2004) are available for determining the moisture content of the paper and paper based products. TAPPI T412 contains the guidelines to be followed for analyzing the moisture content for different paper sample. Small amount of sample is taken in an oven dried weighing container and weighed (W1). Testing time is differently mentioned in TAPPI T412 for different sized specimens e.g. for small specimen the time is 30 min. in the first step of heating. The weight of the specimen taken is more than 2 g and of grammage and less than 224 g/m^2. The time for the first thermal exposure is 1 h. Second step onwards, both the sample is exposed to heat for 1 h until equilibrium weight is reached. The weight of the container along with the sample is noted as W2. The moisture content is calculated using the equation:

$$\text{Moisture, (\%)} = ((W1-W2)/W1)*100$$

Where, W1 and W2 are the original weight of the specimen and weight of the specimen after oven drying respectively.

Coefficient of friction

TAPPI T549 contains the guidelines required to measure coefficient of static and kinetic friction of uncoated printing and writing paper when they are slid against itself by using "horizontal plane method"(Fulleringer and Bloch 2015). TAPPI T548 contains the guidelines for measuring coefficient of static friction for uncoated printing and writing paper by using the inclined plane technique method (McDonald 1999). The determination of coefficient of static friction for packaging material (corrugated and solid fiber board) by inclined plane method is carried out using the methodology given in TAPPI T815 (Vosler 2006). ISO 15359:1999(Kaushik et al., 2015) also contains the regulations for determining coefficient of static and kinetic friction using horizontal plane method.

Taint and odour Robinson sensory test

BS EN1230-2:2009 describes the process to investigate suitability of using paper and paper based product as food contact materials (Nowacka et al., 2018).There is a high chance to transfer taint and odour from paper based food contact materials to food items. Such tainting and odorous chemicals can be identified by using chromatography (GCMS or HPLC).

Inter-fiber bonding and inter-fiber contacts

Cellulose fibers exhibit high swelling attributes in aqueous medium. According to Hirm and Schennach (2015), "They form a soft hydrogel layer on their surface" (Hirn and Schennach2015). The gel layer in turn enables inter-diffusion of fibers among the layers of celluloses. It is believed that "key mechanism of fiber-fiber bonding" is inter-diffusion as it enhances the fiber-fiber contact area available for inter polymer bonding interactions. Fluorescence Resonance Energy Transfer (FRET) microscopy is adopted by Thompson et al. (2008) to investigate the degree of bonding among fibers for softwood pulps (Thomson et al., 2008a). FRET are generally used to investigate the type of interaction between two dye molecules (One is acceptor and the second one is donor molecule). The interaction is called as electrostatic if the distance between two types of dye molecules lies within 100 angstrom. 7-diethylaminocoumarin-3-carboxylic acid hydrazide (DCCH), and fluorescein-5-thiosemicarbazide (FTSC) are the example of donor and acceptor fluorescence dyes respectively. The excitation wavelength of donor dye and acceptor dye are 440 nm and 500 nm respectively. The emission can be detected at 485 and 535 nm, respectively, for the donor and acceptor dye. Here, the interest is to observe the detection of emitted wavelength at 535 nm when excited the test material with 485 nm. Here, two sets of cellulosic fibers are dyed with two different dyes separately. Then the fibers are mixed and subjected to wet pressing or capillary forces. The product is then illuminated with light of appropriate wavelength for the excitation of donor dye molecule and each intersections of the fibers are subjected to this study to obtain fluorescent micrograph.Three fluorescent micrographs are collected with each of the three filter sets which are D, A and F. Here, D, A and F are Donor Excitation and Donor Emission, Acceptor Excitation and Acceptor Emission and Donor Excitation and Acceptor Emission respectively.As a result, if acceptor fluorosence is detected then the differently dyed fibers could be thought of having intermolecular distances less than 100angstrom. The fluorescent micrographs are subjected to analysis by the “Gordon’s 1997 FRETN algorithm” (Thomson et al., 2008b). Except this, AFM is also suggested for the investigation of inter-fiber adhesion (Hirn andSchennach 2015).

Optical property

Optical property of paper and paper based products are measured focussing on (i) brightness, (ii) opacity, (iii) gloss, (iv) whiteness and (v) color.

Brightness is measured using the guidelines given in TAPPI T452 (Anupam et al., 2018) for different products like, white, near-white and natural colored paper, pulp and paperboard. This method involves an instrument which uses 0° viewing geometry and 45° illumination. This measurement is not applicable for paper or paperboard bearing color like yellow or green dyestuff and colored papers which needs to be measured either colorimetrically or spectrophotometrically to attain results.

Opacity is measured using standard TAPPI T425 (TAPPI 2007). Opacity Meter is used to determine the opacity for any paper surface which is the reflectance of paper when it is joined with a white backing is higher than that of paper when joined with a black backing because, in the first case, light transmitted through the imperfectly opaque sheet is largely reflected by the white backing, and a portion of the light is transmitted through the paper at the second instance; thus raises the total reflection. This method utilizes backing or paper backing. Moreover the method for determination of the opacity (paper backing) of paper is specified in ISO 2471 (Stawarczyk et al., 2013). This standard is not applicable for colored paper or boards which contains fluorescent dyes or pigments.

TAPPI T480 contains the guidelines for measuring specular gloss of paper at 75° (15° from the plane of paper). This standard is applicable for coated and variety of uncoated paper. This method is also suitable for low to high gloss paper and for ink films on paper and paper board (Ye at al. 2006).T653 contains the guidelines for determination of specular gloss of paper and paper board at 20° applicable for high gloss papers. This method is used for partial measure of the surface quality and shiny appearance of coated paper (Zwinkels et al., 2018).ISO 8254-1contains the guidelines for determination of specular gloss measured at 75° using convergent beam geometry (ISO 2009)

Whiteness is characterized using the guidelines of TAPPI standard T560 om-05. It is used to measure“ CIE whiteness and white indices” of specimens which are white or nearby white. The entire visible spectrum is involved during measurement of whiteness (Zhang et al., 2018).

The guidelines for determination of whiteness for paper and paper board (with or without fluorescent whitening agent) are also stated in ISO 11475:2017. The values obtained indicated whiteness of the sample when viewed under CIE D65 daylight which is the standard illuminant. The entire visible spectrum is involved during measurement of whiteness. BLAZNIK et al. (2017) reported the change in optical property of paper when exposed to light at 35°C and 65°C. The optical property was measured according to guidelines given in standard ISO 11475:1999. (Blaznik et al., 2017).

MECHANICAL CHARACTERIZATION TECHNIQUES

Various mechanical characterization techniques are adapted to obtain useful information regarding the durability of the moulded articles. Among them, the mechanical characterizations used majorly are discussed below.

Short span compression strength

ISO 9895-2008 contains the guidelines for measuring compressive strength of paper and boards in the machine and cross direction using short-span compressive tester (Adamopoulos et al., 2014). This standard is suggested for boards and paper having grammage between 100-400 g/m^2.

Tensile testing method

Pre characterization procedure is required for conditioning of the product. Here, the product is kept at 20°C and 65% relative humidity. Samples of size 80 mm in length, 25 mm in width, 50 mm in gauge length are used for tensile testing (Curling et al., 2017). The sample is drawn in a uniform rate often sile force until failure. The modulus can be determined using the following equation:

$$modulus = \frac{Force\ applied\ (N)}{width\ (mm) X depth\ (mm)} \Big/ \frac{Deflection}{Span\ length}$$

For the papers the tensile test is carried out as described in the standard ASTM D3039 (De Baere et al., 2009).The extent of reinforcement for the natural fibers can be tested using the same procedure by impregnating the fibers in the resin before the testing. The resin properties can be obtained as per the guidelines given in ASTM D638 (Singh et al., 2014).Dimension of the sample taken is 12.7 mm in width and 170 mm in length.

TAPPI T 494 contains the guidelines for determination of (a) tensile strength (b) stretch (c) tensile energy absorption and (d) tensile stiffness, for all types of paper and paper board (TAPPI 2002). ISO 1924-2 also describes the procedure for determining the tensile properties of paper, board and pulps using constant rate of elongation of 20 mm/min and 100 mm/min respectively (ISO 1924). ISO 1924-2 contain the guidelines used to determine tensile index, tensile energy absorption index, modulus of elasticity, tensile strength, strain at break, tensile energy absorption.

Flexural properties

Three-point loading in an Instron testing machine (Instron) is used to investigate the flexural properties of the product material. 5 kN load cell, in a controlled environment (20°C and 65% RH) is used for flexural characterization. The test span length is taken similar to the gauze length used in tensile testing (Curling et al., 2017). The modulus for bending can be estimated using the following equation:

$$modulus\ (bending) = \frac{Force\ applied\ (N) X Span\ length^3}{4 X Deflection X Breadth X depth}$$

Bending resistance/stiffness

Stiffness and resistant to bending are determined using the method ISO 2493-1992, ISO 5628 (Fellers 1997) in both the cross and machine directions. The stiffness indicates the compression strength and the ability to maintain the shape of a package.

Burst strength

ISO 2758:2014 is used to determine burst strength for paper and paper based products. This is an important test to determine the resistance of a paper sacks and bags against bursting open if dropped or in normal service conditions. Tesfaye et al. (2017) synthesized paper from waste chicken feather. Bursting strength of the synthesized paper was carried out using the guidelines given in standard ISO 2758:2014 (Tesfaye 2017). The bursting index can be estimated using the equation:

$$Bursting\ index = \frac{Bursting\ strength\ (kPa)}{Grammage\ (\frac{g}{m^2})}$$

TAPPI T 403 contains the guidelines for determining the maximum bursting strength of paper and paper products (newsprint, fine paper, packaging paper, bag paper, and printing paper) which can be used if bursting strength lies in the range of 50kPa-1200kPa (TAPPI 2002). TAPPI T807 (Sharma and Garg2017) and TAPPI T810 (TAPPI 2011) contain the methodology for determining the bursting strength of “Paper and liner board” and “corrugated and solid fiber board” respectively. The samples to be tested are conditioned according to the methods given in standard TAPPI T402 (Tappi 2013). The corrugated fiberboard which can be tested using the techniques given in TAPPI T810 must be single walled or double walled type and have bursting strength in the range of 690 kPa to 4825 kPa.

Elmendorf tear resistance

Tear strength is characterized by Elmendorf tear tester by measuring the work done for the specimen under characterization. ASTM D1922-15(Briassoulis and Giannoulis, 2018), ISO 1974-2006(Korean Standards Association 2006)are the standard method used for this testing. This is used for testing the tearing resistance (out of plane) of paper and boards having low grammage provided the tear resistance of the board to be tested falls within the instrument range. The tear resistance of corrugated fiber board cannot be determined using the guidelines of the standard.

Interply bond strength (z direction Tensile strength)

Interply bond strength is determined using the guidelines given in the standard ISO 15754- 2009 (Tausif and Russell, 2012). This is used for testing the bond strength among the fibers present in paper and paperboard. Generally, paper and paperboard made by the vat method is mechanically weaker along the z direction. Hence, it is essential to investigate the interply bond strength along the z direction.

Morphological studies

In case of pulp and paper based products, two types of morphological characterizations are generally preferred. One is the study of fibers present in pulp materials and another one is the surface of the moulded articles made by the pulp. Scanning electron microscope is used to investigate surface morphology. Sample preparation technique is very much essential here on which quality of the image depends. The sample preparation involves fixation of the pulp fibers by mixing it in a mixture of 3% (by volume) of glutaraldehyde and 2% (by volume) of formaldehyde. Time required for fixation is 24 h. Then the sample is washed by distilled water and then is treated with various ethanol-water mixtures with a concentration ranges from 70% to 100% ethanol for 30 minutes in each of the mixture. Then it is subjected to air drying and gold coating subsequently. The images can be taken at 15 KV using SEI detector in normal SEM (Singh et al., 2011). Light microscopy can also be used to investigate the fibers after boiling the pulp fibers in lacto-phenol (Singh et al., 2011). Two types of morphologies are examined for pulp based moulded articles which are bulk morphology and surface morphology. Bulk morphological study is carried out on fractured surface (Either tensile fractured or impact fractured) of the paper and pulp based moulded articles.

APPLICATIONS IN PACKAGING

Paper and paperboard are a popular choice in developing various packages like cartoon, boxes, Tetrapak etc. Use of this type of eco-friendly material increases till huge deposit of plastic waste comes under notice. This type of pulp based materials finds use in developing egg cartoons, hand set packaging as “cushioning consumer durables” (Wever and Twede 2007) Cellulosic fibre is enormously used in packaging of food and beverages. Cellophane is another example which is mostly used as packaging material for food items. Cellulose acetate along with cellulose triacetate are used as rigid wrapping films due to its low gas and moisture barrier properties. “Nature Flex” was marketed by a US based company Innovia Film that was reported to offer “an extremely wide heat seal range, printability, long shelf life and good gas barrier properties” (Abdul Khalil et al., 2016). Billerud Korsnäs from Sweden introduced “Fibre Forms” in the year 2009 as a renewable packaging materials. The “Fibre Forms packaging” was reported to have high elasticity and strength, and its high purity were approved for direct contact with food. Elo Pak, another leading food packaging supplier, produced Pure-Pak cartoons (This consists of a PE layer) for packing the milk, juices etc. (Flisberg et al., 2009). Moreover, cellulose composites have a large potential for the use in corrugated boards. A comparative study for the different pulp materials for this is reported by Tervahartiala et al. (2018).

CONCLUSION

Environmental aspects poses greater interest in using paper and pulp based moulded articles as a replacement of single use plastics for the use as packaging materials. Paper and pulp based materials possess excellent biodegradable characteristics which has made this material more eco-friendly in nature. But this area is not yet well explored. This review paper not only briefs about the detail about such eco-friendly materials, manufacturing technologies, characterization techniques along with the way to recycle it. Moreover, several challenges faced during recycling and their possible outcomes are also discussed in this review article which may become useful in developing future products using paper and pulp materials. In view of the literatures, many important characterization techniques used for characterizing these types of moulded articles are discussed which are helpful to carry out the future research on improving the properties of the products made of pulp.

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