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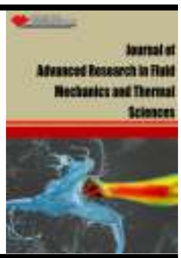

Void and Moisture Content of Fiber Reinforced Composites

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ABSTRACT

In this paper, a review of the void and moisture content studies of fiber reinforced composites for both, synthetic and natural based fibers are presented. The review summarized the research papers in which include experimental and theoretical works that related to the void and moisture content studies. In addition to that, this review paper highlighting a few research studies conducted in literature on the effects of the void and moisture on the mechanical performances of the composite. Few common measurement methods used for the void and moisture determination are discussed here. The aims of this review, mainly to capture the trend ranging from the recent five years back and summarize the various studies and to compare the most common method for the determination of the void and moisture content. This paper is providing a baseline in the selection of the methods for the future work of the author's work with regard to the reduction of the presence of voids and moisture occur during the impregnation process of pre-pregs and their fiber reinforced composites, especially when using natural-based fiber.

1. Introduction

Fiber reinforced composites (FRC) are widely used in lightweight structural applications by the reason of their superior mechanical properties and lightness. Synthetic fibers are extensively used to reinforce polymeric resins in the creation of composite materials due to their advantageous mechanical properties. In the automotive industry, they are also specifically used because composite materials give a higher strength-to-weight ratio. The widespread use of synthetic fiber, however, contributes to environmental and recycling concerns. Natural fibers, therefore, deliver less costly and sustainable fiber resources. Natural fiber reinforced composites (NFRC) are gaining wide popularity due to its potential to be an alternative and environment friendly option to the existing synthetic fiber composites. The advantages of NFRC over the manmade fiber composites are renewable,

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carbon neutral, comparable high mechanical properties, low cost and weight, recyclable, biodegradable and produce organic waste that can be used to generate electricity at the end of their life cycle. They have several benefits over the conventional composites in terms of the high strength, light-weight, high durability and water resistant, chemical resistant, electrical resistant, fire resistant and corrosion resistant properties. In many engineering applications with an extremely wide variety of properties, NRFCs are being used increasingly. Whether derived from plants, animals, or minerals, natural fibers are classified according to their origins. It has been reported that natural fibers been used in various application, such as packing, building, ship, and automotive due to extraordinary properties compared to other synthetic fibers [1]. Plant fibers include leaf fibers (pineapple, sisal, and abaca), core fibers (hemp, jute, and kenaf), grass and reed fibers (wheat, corn, and rice), seed fibers (cotton, kapok, and coir), bast fibers (flax, jute, hemp, ramie, and kenaf), and all other types (wood and roots) [2].

Somehow, manufacturing defects are one of the key variables that deviate from the expectations of the mechanical properties of NFRC. They are described as irregularities that cause the composite properties to deviate from requirements that have been designed. Defects can be classified, based on their location, into matrix, fiber, and interface defects. Fiber defects include fiber waviness and misalignment and broken fibers (due to fiber curving during manufacturing, friction in the textile machine), interface defects include initial fiber/matrix debonding and interlaminar delamination, and matrix defects include incomplete matrix cure, and voids. Voids refer as the regions unfilled with polymer and fibers, are one of the most significant defects [3]. Their significance is due to their considerable effect on a wide range of composite properties and mechanisms leading to failure as well as to their high formation probability in different manufacturing techniques. As a result of their importance, voids are by far the most studied manufacturing defect. Water and moisture absorption of natural fibers have multiple effects, in terms of their properties, morphology, chemical composition, and dimensional stability. The composites made with fibers taken from different relative humidity (RH) environments are expected to behave differently. On the other hand, when the composites are made with dry fibers, these can also absorb moisture in various conditions of humidity. Furthermore, if the composites are immersed under water, their properties are likely to be degraded by absorbing water. Excessive water or moisture can result in the swelling of the fiber that could minimize the mechanical and dimensional properties of the composites as a result of the appearance of micro cracks at fiber-matrix space [4]. The results in the past study showed that the moisture content affected the mechanical properties of the composites. For example, the strength and elongation at break of the jute yarn decreased with a decrease in moisture content [5].

In this paper, a review on the void and moisture content studies of fiber reinforced composites for both, synthetic and natural based fibers are presented. The review summarized the research papers in which include experimental and theoretical works that related to the void and moisture content studies. In addition to that, this review paper highlighting a few research studies conducted in literature on the effects of the void and moisture on the mechanical performances of the composite. Few common measurement methods used for the void and moisture determination are discussed here. The aims of this review, mainly to capture the trend ranging from the recent five years back and summarize the various studies and also to compare and conclude the most common methods for the determination of the void and moisture content. This paper is mainly providing a baseline in the selection of the methods for the future work of the author’s work with regard to the reduction of the presence of voids and moisture occur during the impregnation process of pre-pregs and their fiber reinforced composites, especially when using natural-based fiber.

2. Past Studies on Void Content Investigation

Void also known as porosity is defined as defect that occurs during the manufacturing process and it may play significant role in the mechanical performance of composites. The void content and their morphology may influence the composite properties, especially for the structural laminate of composites, when their presence between the layers, in which randomly generated during processing could resulting in obvious stress concentrator. This situation may lead to the composite delamination and crack initiation before propagating towards laminate failure. Understanding the void formation is important to reduce the void content percentages and their morphology (i.e., shape and size).

The manufacturing processing conditions are one of the factors contributing to the presence of void contents, which then reduce the performance of the composite itself. For example, voids may form during compounding or melt-flow processing, or because of uneven shrinkage during the solidification phase due to thermal gradients (cooling). An increase in the cooling rate results in an increase in the void content of composites. The fiber volume fraction and fiber length in the composites also are the main factors for void or bubble formation. The probability of void formation increases when the composite having higher fiber content and length. Figure 1 shows the extensive work carried out by various researchers from 2015 to 2020, studied the presence of void contents in their composite, which have been fabricated using various processing methods.

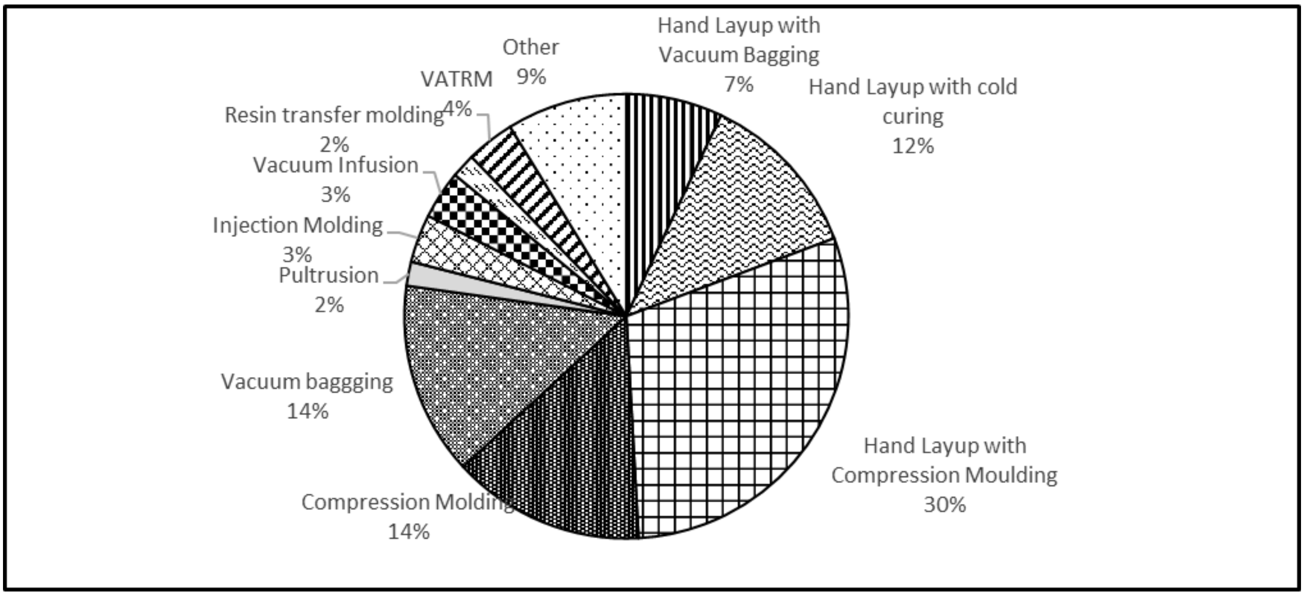

Fig. 1. Research papers on the study of void content of composites fabricated using various methods

In addition to the production technique and processing parameters, matrix material, moisture and solvent content, stacking sequence, and composite thickness are some of the factors that can affect the void content [6]. Air or other volatile compounds may be caught in the material during the incorporation of fiber into the matrix. Due to the fiber spacing and between the laminate and resin-rich regions, micro voids can form in the composite along the individual fibers after curing, which has an adverse effect on the mechanical properties of the composites. Curing and cooling rate of the resin can also responsible for void formation [7].

Due towards the importance of the void characterisation, the void formation, characteristics and effects on mechanical properties in FRC have been extensively studied and summarized by various researchers [3]. They reported that, there are few types of the void morphology and characteristics, mainly the void types and their location of in the both unidirectional and woven-plies composites. In the studies with regard to woven fabric pre-preg and their composites, the main types observed in woven fabrics are (i) micro-void (intra-laminar void), with narrow and elongated shape in fiber direction inside tows, (ii) meso-void with the shape of irregular or spherical in resin-rich regions at tow intersections, i.e. at tow corners, (iii) flat void with pan-cake shape, occurs between the plies, i.e., between the overlying tows of two adjacent plies, and (iv) micro-void, with shape of small sphere exists usually in resin-rich regions.

In the reduction of the void content in the composite final products, the impregnation process and the evaporation of the volatiles are the key factors to be controlled, apart from the type of curing methods used for the composite manufacturing and their process parameters. The manufacturing technologies for composites using pre-pregs commonly with autoclave, Out of Autoclave (OoA) curing and automated pre-preg laying methods, in which the percentage of voids can be reduced mainly, by controlling the pressure and temperature. These issues have been addressed by various researchers as reported by Medhikhani *et al.,* [3] in their extensive review. In the author's work, the schematic diagram of the void formation and their mechanism is carried out as can be seen from Figure 2 below, in understanding the void formation in the Out of Autoclave (OoA) process.

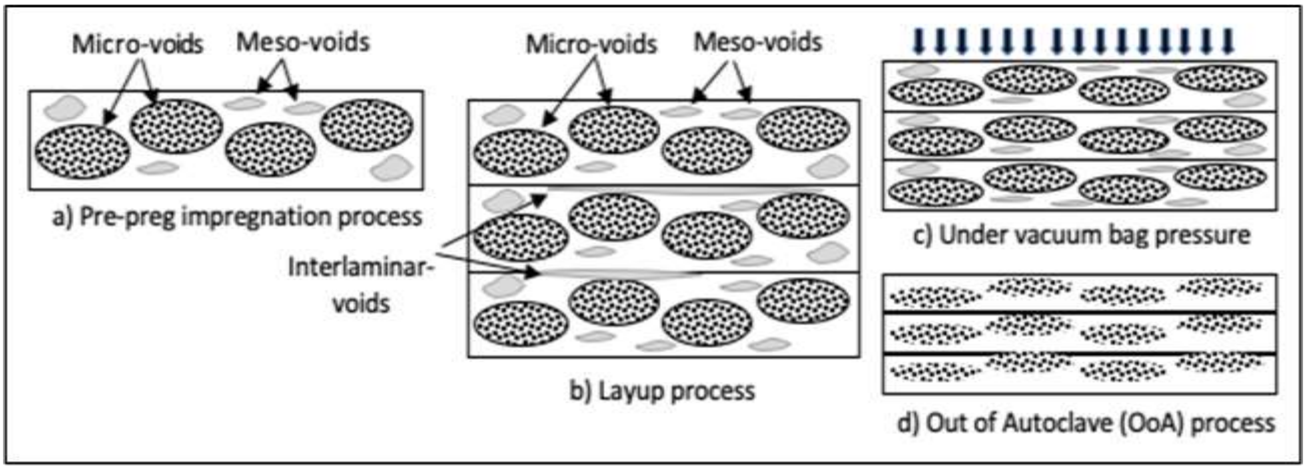

Fig. 2. The schematic diagram of woven fabric cross sectional area view with tows (a) The presence of void formation during pre-preg impregnation (b) Pre-preg layup process (Partially cured B-stage) (c) Pre-preg laminate under vacuum bagging and heat application using Out of Autoclave OoA process for the manufacturing of composites (d) Removal of voids after the Out of Autoclave (OoA) process, with the fully consolidated cured composite laminates

It is importance to extensively monitor the presence of voids in the pre-preg manufacturing and OoA process, therefore this review outlines a few techniques and methods for void content studies that has been summarized with regard to their advantages and disadvantages offered are shown in Table 1. There are few methods either using testing tools or set up of experiments in order to determine the void and using being used widely in various studies, mainly optical microscopy, scanning electron microscopy, microscopic computed tomography, ultrasonic testing or micro-CT and density determination. The methods used, are, either destructive or non-destructive, respectively.

Table 1
A few techniques and methods for void studies and their advantages and disadvantages [3]

Techniques	Measurable parameters	Advantages	Disadvantages	Reference
Density determination	- Void content	- Easy and inexpensive - Relatively quick	- Accuracy dependence on input properties - Destructive - Needing density input - Providing nothing on other void characteristics	[13-14]
Microscopy	- Void content - Size - 2D shape - Location /distribution	- Easy and inexpensive - Relatively quick - Providing 2D morphological output	- Section-biased - Location-biased - Needing multiple analysis - Destructive	[9-15]
Ultrasonic testing	– Void content – Planar size – Planar location/ distribution	– Precise – Non-destructive – Possible for in-service inspection – Portable	– Needing a coupling agent (Improvement under progress) – Only applicable to flat and smooth surfaces (Improvement under progress) – Time consuming (improvement under progress) – Providing limited morphological output	[16–18]
Micro-CT	– Void content – Size – 3D shape –Location /distribution	– Relatively accurate – Providing full 3D analysis – Semi non-destructive	– Needing small samples – Location-biased – Time consuming – Costly	[9-13-19]

Ultrasonic testing, with mature and standardized industrial testing apparatus and procedures, remains the main industrial instrument for assessing the void content and making a reject/accept decision. However, ultrasonic inspection does not provide precise information on the morphology of the void, the size of the individual voids (and the statistical distribution of these parameters), and the spatial distribution of the voids. Micro-CT is the most advanced and reliable, albeit costly, tool for accurate and detailed void observation and investigation. Methods for quantifying micro-CT images are emerging, but they are still an active subject of research and will likely mature in the next few years. High fidelity micro-CT is restricted to small-size specimens. There are various kinds of microscopes that can be used for imaging material cross-sections, such as optical and Scanning Electron Microscope (SEM). Commonly, the SEM method is widely used researchers to study the morphology especially for the study of fiber-matrix interfacial adhesion [8]. The most notable and critical difference between the two-microscopy images is the superior contrast between void and matter (fiber and matrix) of the SEM image in comparison with the optical image [9]. A study found that the value of void content measured through image analysis (performed on microscopy images of cross-sections) is higher than that obtained through the density determination technique [10].

Density testing technique that is popularly used by the researcher is a non-destructive and non-visual technique that is able to calculate the overall void volume fraction within a specimen, providing accurate material properties and mass fraction values. The density of resin and composite can be measured through water buoyancy by Archimedes principle (ASTM D792), the density-gradient technique (ASTM D1505), and a direct measurement of weight and volume. The density of the fibers is to be measured through water buoyancy (for the fibers that absorb water). The matrix and fiber contents (weight and volume percentage) can be measured by the digestion or ignition of the matrix (ASTM D3171 and ASTM D2584) or by microscopy followed by image analysis methods. There are few researchers conducted analysis uses digital image processing, Matlab, and Microsoft Excel application to obtain the value of void fraction [11]. Accurate void content measurement requires accurate knowledge of the physical properties of the constituents, namely fiber and matrix. The most advanced and reliable, expensive, tool for precise and detailed observation and investigation of voids is micro-CT or also known as X-ray micro-CT. Micro CT technique is a nondestructive virtual slicing technique. Methods of quantification of the micro-CT images are emerging, but still are an active research subject and will probably be matured in the coming few years. High fidelity micro-CT is limited to specimens of small sizes [12]. With the capability to focus the X-ray beam to a-few micrometers spot size, X-ray micro-CT emerges as the most accurate technique available for 3D evaluation of microscopic features, including voids inside FRCs. Still, the accuracy of the results depends largely on the image processing techniques that are employed for detection of voids. In some studies, synchrotron micro-CT is used for high-resolution analysis of voids [3].

With aim of reducing the void content percentages, in the various work related to the manufacturing of composites, most of them, emphasizing the measurement of the void content in their works. A tabulated summary of research papers investigated on the void content analysis using direct measurement method is shown in the Figure 3. It can be significantly observed that the most common direct measurement method to study void content is using Scanning Electron Microscopy (SEM), followed by the use of the Micro-computed tomography and linear optical microscopy and image analysis such as Image J software to estimate the void content percentages. Another method, which is using an Ultrasonic Scanning technique are getting less attention among the researchers. It may be due to the higher cost and more complicated preparation of samples, even though this method can be considered giving a comparable and reliable finding.

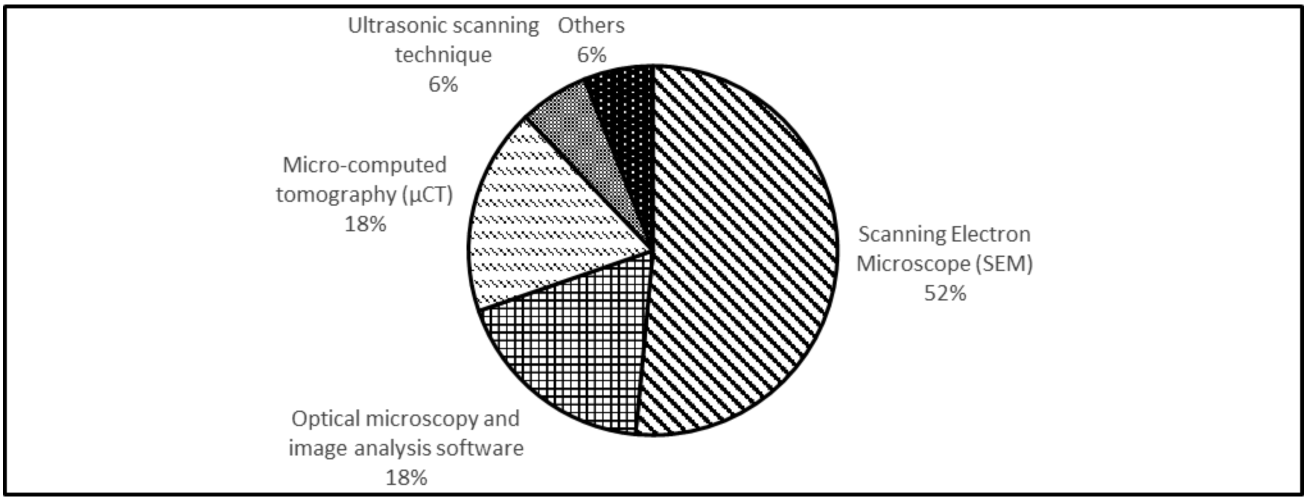

Fig. 3. Research papers on the study of void content of composites using various tools/methods [12-14-37-44-47-68]

Apart of the direct measurement using the common tools/equipment of the experimental study, some of the researchers also conducted in direct measurement from the void content parameter to determine and estimate the void content percentage in the composites. A summary of research papers investigated on the void content studies using in direct measurement methods is shown in the Figure 4. It can be observed that most of the researchers are carried out the void measurement of the void content parameter such as void volume fractions and also density measurement, such as using ASTM 2734-70 and ASTM D3171. There are also very few studies such as conducted recently by Monticeli *et al.,* [20]. using response surface methodology (RSM) equation, in evaluating the 3D void formation through a statistical approach such as ANOVA and Weibull model derived from the basic concept of void volume fraction measurement, with the aim of proposing a method to unveil the role of voids in fiber composite laminates manufactured via resin transfer molding [20].

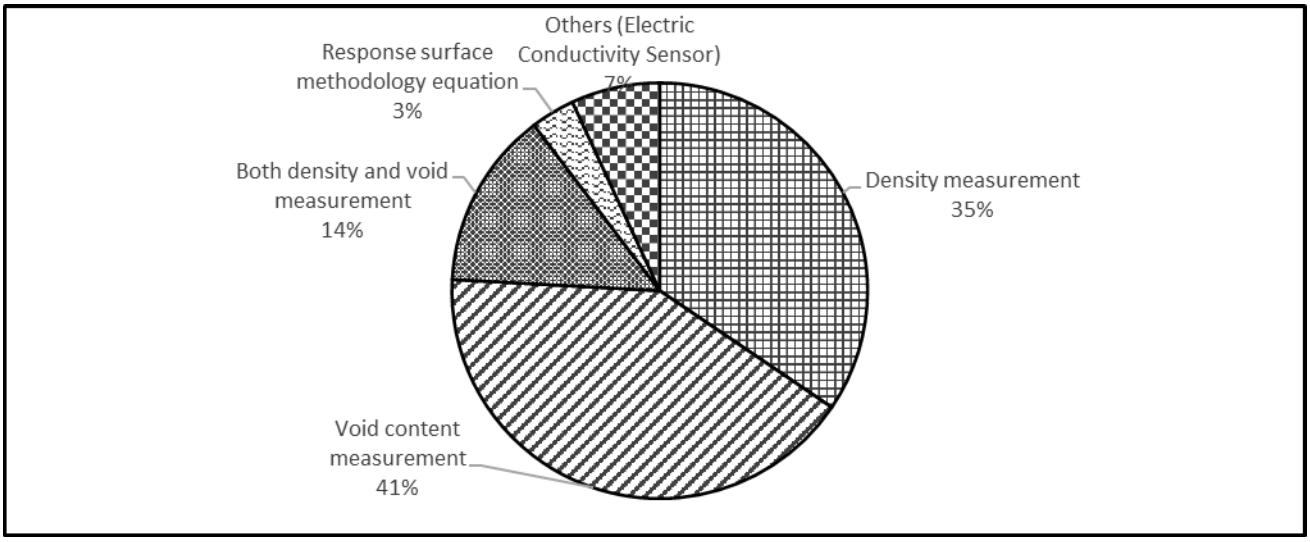

Fig. 4. Research papers on void content studies using the measurement (equation) of some parameters of the void content [14-20-30-37-52-58-62-64-69-82]

3. Past Studies on Moisture Content Investigation

Another important problem with NFRCs, besides void presence, is moisture content. The moisture existence further uptake led to degradation issues of composite laminates. Moisture content of materials is the amount of water contained in the systems that has significant effect on water absorption and fiber swelling behaviour [21]. As reported by Wandowski *et al.,* [22] the moisture update could accelerate to reduce fatigue life, particularly on impacted laminates. Moisture is typically either characterized in terms of relative humidity in surrounding the specimen under investigation or specifically the moisture content (relative moisture mass) in the specimen itself. The moisture content offers a direct intrinsic measure of hygroscopic state of the material, although it is not always practical to quantify experimentally [23]. The presence of moisture influence that the fiber-matrix integrity for long-term performance of composites. Therefore, various experimental works, particularly related to natural-based fiber, also include the study on the measurement of moisture absorption behaviour.

With the presence of void formations in the OoA process, commonly in vacuum bagging process, which resulting from the processing stage shown in Figure 2, lead to the introduction of the moisture content in the composite systems. Apart from the air entrapment either inside tows or between plies during the vacuum bagging curing and OoA, moisture in the resin is another problem. The main volatiles within a pre-preg is water, with other volatiles such as alcohol, acetone, and ethanol. It is reported that if the water vapor pressure exceeds the hydrostatic resin pressure during cure, the moisture-rich voids can exist. Again, it is a need to control the moisture content in the pre-pregs, for example by having a conditioning procedure prior laying up and cure. The mechanism of the formation of moisture content during pre-preg impregnation and their vacuum bagging with OoA processes, affect the properties of the NRFCs is illustrated from Figure 5. Moisture absorption into the pre-pregs and their composites is by three main mechanisms: i) diffusion of water molecules through the micro-gaps between matrix chains; ii) capillary transport of water molecules into the gaps and flaws at the interface between the fibers and the matrices due to incomplete wetting and impregnation; and iii) transport of water molecules through micro-cracks in the matrix, formed during the fabrication process or arising from swelling fibers. After the fiber-matrix bonding is broken, the fibers fail to withstand the load adequately. The broken interface resulting and localizing matrix cracking. This cracking propagates and then lead to larger fiber fracture, delamination, or both. As the consequences, it may lower the mechanical properties of the NFRCs.

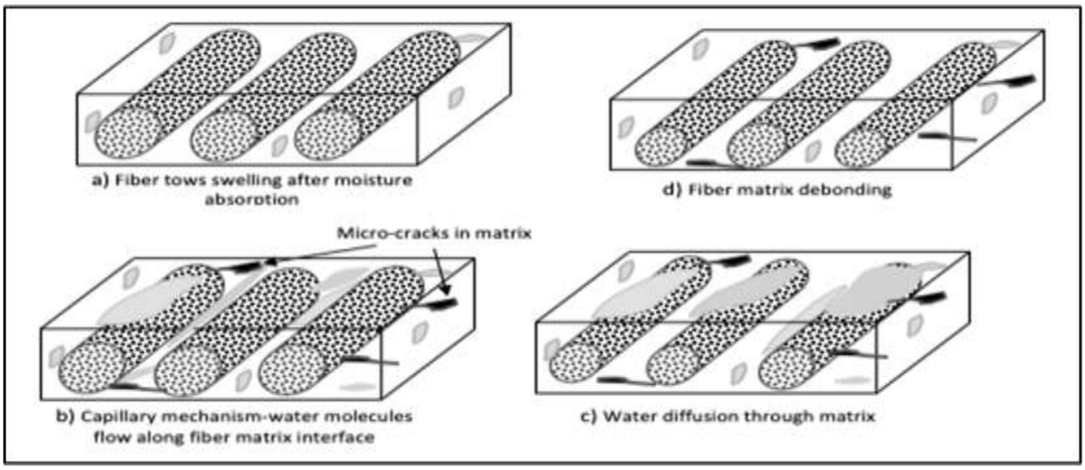

Fig. 5. Moisture content (plain grey in colour) mechanism during the pre-preg impregnation and Out of Autoclave (OoA) curing process

A summary of research papers investigated on the moisture content analysis studied on the common experimental moisture content studies using various tools or methods is shown in the Figure 6. It can be observed that the most common method is mainly from the measurement of the weight fraction and followed by drying oven method, that is a thermogravimetric method (loss on drying) in which the sample is dried for a period of time at constant temperature. The moisture content is determined by weighing the sample before and after drying. There are few improvised methods have been limitedly used to measure moisture absorption, namely as Electromechanical impedance (EMI) method [22] and Impedance spectroscopy [24]. Apart from that, there are few researchers used method with the determination by using absorbed moisture equation such as using ASTM D 5229/D 5229M, to measure moisture uptake, and also few works using Fick's diffusion laws.

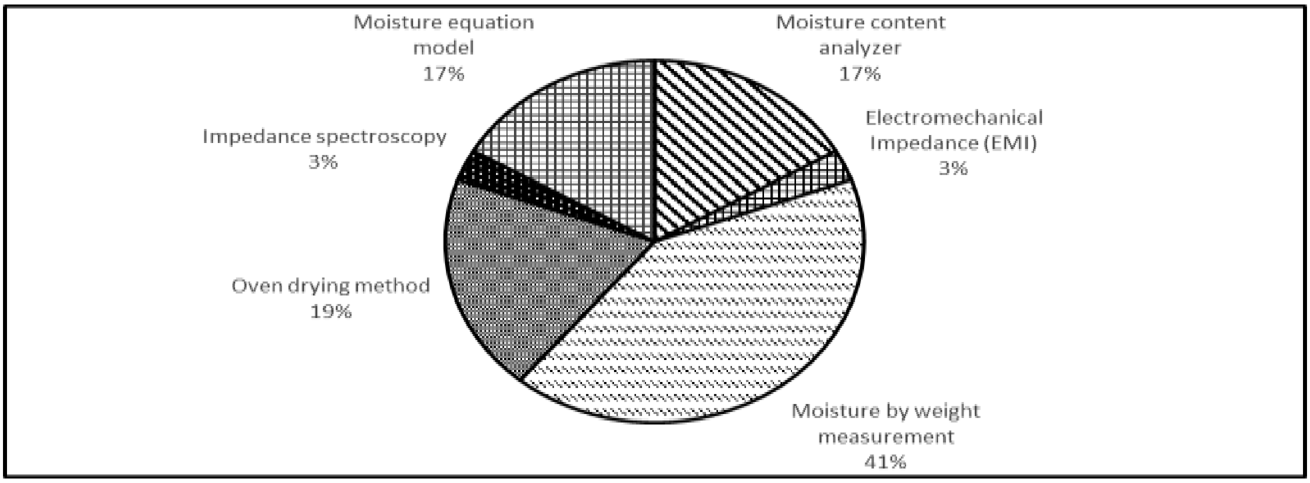

Fig. 6. Research papers studied on the common experimental moisture content studies using various tools or methods [5-22-24-29-33-41-71-81-83-106]

4. Effects of Void and Moisture Content on Mechanical Properties of Composites

The properties of NFRC are different to each other according to previous studies, because of different kinds of fibers, sources, and moisture conditions. As reported, the performance of NFRC relies on some factors, like mechanical composition, micro-fibrillar angle, structure, defects, cell dimensions, physical properties, chemical properties, and also the interaction of a fiber with the matrix [25]. A tabulated summary of research studies conducted on effects of void and moisture contents on mechanical performance of composites is shown in Table 2. The reported research as shown in Table 2 obviously highlighting that the presence of void and moisture content in the fiber reinforced composite, mainly in NFRC, is a problematic issue which need to be addressed thoroughly.

Table 2
Research papers studied about the effect of moisture and void content studies on mechanical performances of composites

Author (Year of Publication)	Type of fibers used	Mechanical Properties
(Chaudhary, 2020) [29]	Jute, hemp, and flax/epoxy	Hardness, Tensile testing, flexural testing and morphological analysis
(Karimzadeh, 2020) [30]	Pineapple leaf fiber (PALF) and glass fiber	Flexural and tensile properties
(Binu Kumar, 2020) [31]	Banana fiber and neem fibers	Tensile strength
(Venkatesha, 2020) [32]	Bamboo and E-glass woven fabric	Thickness swelling test, flexural and tensile test.
(Moudood, 2019) [33]	Flax fiber	Tensile strength, modulus and strain at failure
(Moudood, Rahman, 2019) [34]	Flax fiber	Mechanical testing and interfacial strength
(Moudood, Hall, 2019) [33]	Flax fiber	Tensile properties and flexural modulus
(Ouarhim, 2018) [35]	Few types of natural fibers	Static tensile and dynamic mechanical analysis
(Kini, 2018) [36]	Glass fiber	Tensile and flexural properties
(Di Landro, 2017) [14]	Carbon fiber	Tensile and compressive tests, short beam shear Tests (interlaminar strength)
(Raghavendra, 2017) [37]	Jute fiber	Tensile and flexural properties
(Liu, 2016) [38]	Carbon fiber	Interlaminar shear strength (ILSS), compression strength, fatigue strength and flexural strength
(Chaichanawong, 2016) [39]	Glass fiber	Tensile test, Flexural test & microstructure analysis
(Alzamora Guzman & Brondsted, 2015) [40]	Glass fiber	Single-fiber tensile test, tension tests & V-notch shear tests
(Munoz, 2015) [41]	Flax fiber	Tensile test, flexural test and morphological analysis
(Berthet, 2015) [42]	Wheat straw fibers	Tensile properties
(Li, 2015) [43]	Flax fiber	Tensile and interlaminar shear properties
(Islam, 2014) [44]	Flax fabric	Tensile test, thermogravimetric analysis
(Dong & Takagi, 2014) [28]	Cellulose nanofibre	Flexural properties
(Perez-Pacheco, 2013) [45]	Carbon fiber	Acoustic emission (AE) measurements & tensile testing
(Hernandez Rueda, 2013) [46]	Carbon Fiber	Interlaminar shear strength, low velocity impact and plain compression

Various quantitative studies of the effect of voids and moistures on the mechanical properties of fiber/resin composites have been carried out and one of major findings as reported that, regardless of resin, fiber type, and fiber surface treatment, the interlaminar shear strength of a composite decreases about 7% for each 1% of void, respectively [26]. The void content in NFRC is a concern because of the voids inherent in natural fibers, which affect the transverse failure of the composites [27]. The studies also concluded that the stiffness or decrease in strength is a linear or second-order polynomial function of the void content. During the injection of the fibers into the resin, air or other volatile substances may be trapped within the composites and then, after curing the composites, micro-voids can form. These micro-voids create poor mechanical properties, which result in sudden failure of the composites. The shapes of the voids can be changed from spherical to elongated, and the void content can be decreased by a post-extrusion hot-drawing approach. An author assessed the effect of void content on the flexural properties of a vacuum-treated random-oriented wood pulp fiber/ starch composite manufactured using hot pressing [28]. Another significant finding from the review also showed that, the presence of voids may increase linearly with the different type of manufacturing processes to produce samples [19].

The characteristic of lignocellulose fibers as hydrophilic and moisture absorbing elements, leads to the presence of moisture in NFRCs. A large number of hydrogen bonds exist between the macromolecules in the cell wall of the plant fiber. The hydrogen bond breaks as moisture from the atmosphere comes into contact with the fiber, and hydroxyl groups form new hydrogen bonds with water molecules. The principal access to the penetrating water becomes the cross-section of the fiber. As a consequence, swelling within the matrix occurs when hydrophilic fiber is reinforced with hydrophobic resin fiber. This induces weak fiber-matrix bonding, dimensional instability, matrix cracking, and poor composite mechanical properties. Therefore, the removal or at least reduce the moisture from fibers is an essential step before and during the preparation of composites.

Currently, there is a common opinion that the mechanical properties of NFRC products are declining as the void and moisture content increases. Therefore, most of the void analysis studies published have concentrated on attempts to link the mechanical properties of NFRC products to the content of voids. It also should be noted here that it is far from sufficient to attempt to associate void content with some mechanical properties of NFRC products alone. It is quite clear that the processing parameters simultaneously influence the distribution, position, shape and size of the void during output. Each of these parameters has a different effect on NFRC products' mechanical properties. Therefore, the increasing trend of the studies on the fiber reinforced composite, are continuously being carried out in order to get the improvement with regards to their mechanical performance. This short review is intent to has a snapshot of the recent trend which showing this extensive attention given by researchers, world-widely, significantly not limited to the one as listed in this review.

5. Conclusions

As for the conclusion, a review on the void and moisture content studies of fiber reinforced composites for both, synthetic and natural based fibers has been presented. This review, has captured the trend ranging from the recent five years back and summarized the various studies. The most common method for the determination of the void and moisture content has been compared and concluded in providing a baseline in the selection of the methods for the future work of the author's work with regard to the reduction of the presence of voids and moisture occur during the impregnation process of pre-preg and their fiber reinforced composites, especially when using natural-based fiber.

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