

**Effect of resin system on the mechanical properties and water absorption of kenaf
fibre reinforced laminates**

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ABSTRACT

The objective of this study is to compare the mechanical and water absorption properties of kenaf (*Hibiscus Cannabinus L.*) fibre reinforced laminates made of three different resin systems. The use of different resin systems is considered so that potentially complex and expensive fibre treatments are avoided. The resin systems used include a polyester, a vinyl ester and an epoxy. Laminates of 15%, 22.5% and 30% fibre volume fraction were manufactured by resin transfer moulding. The laminates were tested for strength and modulus under tensile and flexural loading. Additionally, tests were carried out on laminates to determine the impact energy, impact strength and water absorption. The results revealed that properties were affected in markedly different ways by the resin system and the fibre volume fraction. Polyester laminates showed good modulus and impact properties, epoxy laminates displayed good strength values

and vinyl ester laminates exhibited good water absorption characteristics. Scanning electron microscope studies show that epoxy laminates fail by fibre fracture, polyester laminates by fibre pull-out and vinyl ester laminates by a combination of the two. A comparison between kenaf and glass laminates revealed that the specific tensile and flexural moduli of both laminates are comparable at the volume fraction of 15%. However, glass laminates have much better specific properties than the kenaf laminates at high fibre volume fractions for all three resins used.

Keywords: A. natural materials; B. laminates; E. mechanical

1 Introduction

The advantages of natural fibre reinforced composites in comparison to those reinforced by glass fibre include enhanced energy recovery, carbon dioxide sequestration, low cost, low density, high toughness, acceptable specific properties and ease of separation [1 - 3]. In addition, they cause less dermal and respiratory irritation than glass fibres.

Natural fibre reinforced composites are primarily used as low cost materials that have useable structural properties [1]. They have been used in applications such as automotive interior linings, upholstery stuffing, egg-boxes and electronics packaging [4]. Thus, the use of natural fibres in commercial composites has been limited to the reinforcement of non-structural components.

Surface treatments [1, 4] are often used to improve the properties of natural fibre reinforced composites by bridging the gap in compatibility between the hydrophilic fibres and the hydrophobic matrices. Natural fibres can be modified either by physical or chemical means. Physical treatments change the structural and surface properties of the fibre and thereby influence their mechanical bonding to polymers [4]. Chemical

methods of altering natural fibres involve introducing a material compatible with both the fibres and the matrix [4]. This material can have roles such as increasing wettability of fibres, eliminating weak boundary layers or forming bonds between the fibres and the matrix. A disadvantage of physical and chemical treatments is that they add an extra stage in the manufacturing process which increases complexity and / or expense.

It is possible to alter the properties of natural fibre reinforced laminates without the need for physical or chemical treatments by using different resin systems since they have differing levels of compatibility to natural fibres. A number of common resin systems are available for the production of fibre reinforced laminates and these include polyesters, vinyl esters and epoxies. All have different properties of strength, modulus, adhesion, cure shrinkage and water absorption [5 - 7].

Polyesters are the most widely used resins for fibre reinforced plastics [6, 7]. They are inexpensive, easy to use and have good mechanical properties such as impact resistance but their disadvantages include poor adhesion and high cure shrinkage [6, 7]. Epoxy resins have excellent mechanical properties, low cure shrinkage, excellent adhesion and compatibility with many fibres and other materials. Epoxy resins are, however, generally expensive and can be sensitive to moisture [5 - 7]. Vinyl esters constitute a middle ground between polyester and epoxy resins and represent a compromise in cost and mechanical properties.

The effects of using different resin systems in the resin transfer moulding (RTM) [8] of non-woven flax fibre reinforced composites has been investigated by Joffe et al. [9]. These investigators conducted tensile and fracture toughness tests on flax fibre reinforced laminates made using three polyesters, a vinyl ester and an epoxy. These were compared to glass fibre reinforced polypropylene laminates of equal density. This investigation showed that one of the three polyesters, the vinyl ester and the epoxy

produced flax laminates with comparable properties to those of the glass fibre reinforced polypropylene laminates.

Several investigations [10 – 14] have used polyester resins to produce non-woven natural fibre reinforced laminates. Oksman [3] used epoxy resin and unidirectional flax fibres as it was expected that this would result in good bonding. Rodriguez et al. [15] used vinyl ester resin and alkali-treated, bi-directional jute fibre mats to manufacture laminates by RTM. However, the results of these works [3, 10 - 15] cannot be compared since they all use either different fibres or weaves.

It is clear that a number of natural fibre types can be used to reinforce laminates. However, kenaf offers advantages as a fibre crop including fast growth in a variety of climatic conditions and subsequently fast accumulation of carbon dioxide [16]. Kenaf is grown in a number of countries around the world such as China, India, Indonesia and Thailand [17]. Cultivation and processing of kenaf has recently also commenced in South Africa. According to Zampaloni et al. [18], kenaf fibres cost around 0.40 to 0.55 US dollars per kilogram. Apart from use in natural fibre reinforced composites, kenaf is also being used in particle board, geotextiles and paper production [17].

The objective of the current paper is to systematically compare the mechanical and water absorption properties of non-woven kenaf fibre reinforced laminates made using a polyester, a vinyl ester and an epoxy resin. The kenaf fibre is untreated. In addition, the mechanical properties of kenaf fibre reinforced laminates are compared to those of glass fibre reinforced laminates on the basis of specific mechanical properties. The investigation was partially motivated by the need obtain answers to two important questions:

- Could the mechanical properties of untreated kenaf fibre reinforced composites be improved by replacing the inexpensive polyester or vinyl ester resin with a moderately expensive epoxy resin?
- Could the use of different resin systems be considered as an alternative to potentially expensive surface treatment of fibres?

Since it is known that an epoxy resin generally bonds better than a polyester resin, the answer to the above questions are not very clear in the absence of quantitative data.

While Joffe et al. [9] has carried out a similar investigation, it was for flax fibre composites and no results are available for kenaf fibre reinforced composites.

2 Experimental

2.1 Materials

Sustainable Fibre Solutions of South Africa donated the non-woven kenaf fibre mats used in this investigation. The fibre mat has an areal density of 450 g/m^2 . Glass fibre chopped strand mat (CSM) with an areal density of 350 g/m^2 was used to produce glass laminates for comparison.

Resins were chosen for their suitability to the RTM process. The polyester resin (Scott Bader Crystic 1141) is an orthophthalic unsaturated polyester resin. It was supplied with Crystic Accelerator E and Andonox KP-9 catalyst (M-200 catalyst). The vinyl ester resin used was Derakane Momentum 411-350 epoxy vinyl ester resin. The accelerator used with the vinyl ester was cobalt naphthenate (1 % concentration) and the catalyst was Norox MEKP 925. The epoxy resin comprised SP Prime 27 Epoxy Resin with SP Prime 20 Slow Hardener. Typical properties of the three resin systems were supplied by the manufacturers and can be found in Table 1.

2.2 Production of laminates

An RTM machine designed and built by the principal investigator was used to produce the polyester laminates. The mixing ratios for the epoxy resin fell outside the capabilities of the RTM machine. As a result, this resin had to be manually mixed which caused air bubbles to be introduced. The vinyl ester resin also produced bubbles when the catalyst was introduced. In order to remove the bubbles, the vinyl ester and epoxy resins were first degassed and then injected into the mould using a pressure pot. Accelerator and catalyst were mixed into the polyester resin in a ratio of 1.5 % by mass each. For vinyl ester resin, accelerator and catalyst were mixed into the resin in a ratio of 0.3 % and 1 % by mass respectively. Hardener was mixed into the epoxy resin in a ratio of 28 % by mass.

The mould consists of three mild steel plates; an upper mould plate, a lower mould plate and a spacer plate. The spacer plate has a cavity and an o-ring groove machined into it. The cavity of the mould is sized to produce laminates measuring $280 \times 260 \times 4$ mm. Resin is injected into the mould through a single inlet port located in the centre of the lower mould plate and exhausted via four ports located in the upper corners of the mould cavity. Care was taken to cut the rectangular fibre mats with the same orientation relative to the fibre roll. This was because the mat was suspected to be directional as found by O'Dell [10] for jute fibre non-wovens. Laminates were produced with fibre volume fractions of 15 %, 22.5 % and 30 %. Fibres were dried at 55°C for three hours prior to injection by drawing warm air through the mould.

All laminates were produced with the mould heated to 50-55°C except for glass fibre reinforced vinyl ester laminates. These were produced with the mould at room temperature to avoid the need for an inhibiting agent (2,4-pentanedione) which was found to react with the sizing agent on the glass fibres and make the gassing problem

worse. A temperature of 50-55°C was chosen for rapid but controllable processing. An injection pressure of 250 kPa (gauge pressure) was used for 15 % and 22.5 % fibre volume fractions and 350 kPa was used for fibre volume fractions of 30 %. A vacuum assist pressure of 40 kPa (absolute pressure) was used for all laminates. The injection pressure of the glass fibre reinforced vinyl ester laminates was changed to 0 kPa, 100 kPa and 200 kPa for 15 %, 22.5 % and 30 % fibre volume fractions respectively due to problems with resin flow in the cold mould. The vacuum assist pressure remained unchanged. Following complete mould filling, resin was allowed to run until no bubbles were observed in the exhaust lines. The vacuum was then released and the resin injection line shut-off and disconnected. All laminates were post-cured according to the instructions of the respective resin manufacturer.

3 Testing

3.1 Standards and methods

Tensile and flexural tests were conducted in accordance with ASTM D638 [23] and ASTM D790 [24] respectively on a JJ Lloyd tensile testing machine.

Impact tests were conducted in accordance with ASTM D256 [25] for in-plane Izod impact tests using an instrumented impact machine. Instrumented impact tests allow for the determination of total impact energy, initial impact energy (energy absorbed before the onset of failure), propagative impact energy (energy absorbed between the onset of failure and ultimate failure) and impact strength.

A JEOL 840 scanning electron microscope (SEM) was used to examine the failure surfaces of the impact specimens. Carbon and gold-palladium coatings were applied to the specimens to increase their conductivity.

Water absorption tests were conducted in accordance with ISO R62 [26]. The standard was modified so that measurements of mass and dimension were first taken after 1 hour and then again after a further 23 hours instead of just after 24 hours. Specimens were subsequently measured at various time intervals for four weeks.

Five specimens were used to calculate results of the tensile, flexural and impact tests. Three specimens were used for the water absorption tests. These numbers of specimens are in accordance with the relevant standards.

3.2 Identification of Laminates

Laminates are identified using a coding system. Each laminate is identified using three sets of characters. The first set indicates the resin system: UP (unsaturated polyester), VE (vinyl ester) and EP (epoxy). The second set indicates the type of fibre used: AD (air dried kenaf fibre) or GE (undried glass fibre). The third set indicates the temperature of the mould: H (heated) and NH (not heated). As an example, VE-AD-H stands for: vinyl ester resin, air dried kenaf fibres and heated mould.

4 Results and discussion

The results of mechanical testing are presented in two ways. The first provides a direct comparison of the performance of laminates made using the different resin systems. The second allows for comparison of specific mechanical properties which were determined by dividing the relevant property by the density of the laminate. The latter was calculated using the following equation:

$$\rho_l = \frac{m_l}{\left(\frac{m_l - m_f}{\rho_m}\right) + \left(\frac{m_f}{\rho_f}\right)} \quad (1)$$

where ρ_l is the laminate density, m_l is the laminate mass, m_f is the mass of the fibres, ρ_m is the density of the matrix and ρ_f is the density of the fibres.

In the interest of brevity, kenaf fibre reinforced laminates will henceforth be referred to simply as laminates. Wherever the possibility of confusion arises, however, a full description will be provided.

4.1 Tensile tests

Fig. 1 shows the measured tensile strengths of the different laminates. Error bars in this figure and all subsequent figures correspond to one standard deviation from the mean. It is evident that both the vinyl ester and epoxy resin produce laminates with higher tensile strength than those made using polyester. This is in agreement with the findings of Joffe et al. [9] for flax fibres. It is also apparent that none of the tensile strengths measured at any fibre volume fraction exceed the strengths of the unreinforced resins which are reported by the manufacturers in Table 1 to be 69 MPa, 86 MPa and 73.3 MPa for the polyester, vinyl ester and epoxy resins, respectively. This result is in contrast to those of Rouison et al. [12] who found that hemp laminates with fibre volume fractions greater than 11% increased the laminate strength. The reduction in strength is probably due to the inclusion of defects such as voids and poor interfaces during manufacturing of the reinforced laminates. Even though the unreinforced epoxy resin has a tensile strength that is similar to that of the polyester and lower than that of the vinyl ester resins, epoxy laminates have, on average, the highest tensile strength. This is a good indication that the epoxy bonds better with the fibres than the other resins. The higher tensile strength of the vinyl ester laminates in comparison to the

polyester laminates is believed to be a result of the high strength of the unreinforced resin but it could also be an indication of improved bonding between the fibres and the matrix.

Fig. 2 presents the tensile modulus. It can be seen that the modulus increases with fibre loading. In addition, with the exception of epoxy resin at 15% fibre volume fraction, the values of modulus are higher than those of the unreinforced resins (3.8 GPa, 3.2 GPa and 3.5 GPa for the polyester, vinyl ester and epoxy, respectively, as shown in Table 1). These results are in agreement with the findings of Rouison et al. [12] and Joffe et al. [9] who found similar trends for hemp and flax reinforced laminates, respectively. The latter authors also found that epoxy laminates had higher modulus than either polyester or vinyl ester laminates. In the current work, however, for each fibre volume fraction considered, the tensile modulus of the epoxy laminates is the lowest. This result is unexpected considering that the results of tensile strength indicate that the epoxy bonds to the fibres better than polyester and vinyl ester resins.

4.2 Flexural tests

Results for flexural strength are presented in Fig. 3. It can be seen that the flexural strength of all resin systems increases with fibre volume fraction as found by Rouison et al. [12] and Sèbe et al. [13] for hemp reinforced polyester. It is also evident, however, that the flexural strength of both vinyl ester and epoxy laminates do not show as much improvement as the polyester laminate when the fibre volume fraction is increased from 15% to 30%. A possible explanation is the presence of voids brought on by the need for vinyl ester and epoxy to be mixed by hand and degassed in a pressure pot instead of using the RTM machine. In addition, since stress concentrations due to voids increase with increasing fibre volume fraction, the presence of voids plays a larger role at higher

fibre volume fractions than at lower fibre volume fractions. Fig. 4 shows examples of typical SEM micrographs where voids can be seen in the vinyl ester and epoxy laminates. Although some voids were found in polyester laminates, these were very rare and so a typical micrograph without voids is presented.

Flexural modulus is presented in Fig. 5. Unlike the tensile modulus results, the resin type seems to have little influence on the flexural modulus for each of the three fibre volume fractions considered.

4.3 Impact tests

Fig. 6 presents results from the impact tests. The division in each bar in Fig. 6 indicates the separation between initial impact energy and propagative impact energy. The sum of the two represents the total energy absorbed. It can be seen that the impact energy strongly depends on the resin system. Polyester laminates absorb the most energy before failure while the epoxy laminates absorb the least for all fibre volume fractions. A number of authors [13, 27 - 30] have observed a reduction in impact energy with increased fibre-matrix adhesion. One can therefore conclude that the epoxy resin bonds better with the fibres than the other two resin systems. This conclusion is reinforced when the propagative energy is considered. It is evident that the propagative energy of the epoxy laminates is slightly lower than that of the polyester laminates for each of the three fibre volume fractions. The lower propagative energy of the epoxy laminates is an indication of less fibre pull-out and thus better adhesion between fibres and matrix than in the laminates made using the other resin systems.

The SEM micrographs of impact failure surfaces of composites for 22.5% fibre volume fraction are presented in Fig. 7. It can be seen that the polyester laminates fail by fibre pull-out, the epoxy laminates fail by fibre fracture and the vinyl ester laminates

fail by a combination of both fibre pull-out and fibre fracture. It can also be observed that the epoxy laminates displays a cleaner failure surface than those made with polyester or vinyl ester resins. This perhaps explains the relatively poor impact strength of epoxy laminates displayed in Fig. 8 since a clean failure surface is an indication of short fibre pull-out lengths and good fibre matrix adhesion. The short fibre pull-out lengths result in reduced energy absorption as suggested by van den Oever et al. [27].

Impact strength results are presented in Fig. 8. Similar to impact energy, impact strength is also strongly affected by the resin system. Impact strength is highest for polyester laminates and lowest for epoxy laminates. According to van den Oever et al. [27], a consequence of shorter fibre pull-out length is that the matrix deformation zone is smaller. Concentration of the strain energy into a smaller region causes the apparent impact strength to be reduced in epoxy laminates.

4.4 Specific properties

A comparison of specific tensile strength of kenaf fibre laminates with those made of glass fibre is presented in Fig. 9. It can be seen that the specific tensile strength of the glass laminates is greater than those made of kenaf for each of the three fibre volume fractions. In addition, the glass laminates display a greater rate of increase with the fibre volume fraction than those made of kenaf.

Fig. 10 presents the results of specific tensile moduli. It is evident that kenaf fibre reinforced laminates made using all three resins are comparable to the glass fibre laminates at 15 % fibre volume fraction. At 22.5 % fibre volume fraction, the kenaf fibre polyester laminate is better than the glass equivalent. At 30 % fibre volume fraction, all of the kenaf fibre laminates have specific tensile moduli lower than those of

the glass equivalents. In addition, the resin system seems to have a significant influence on the specific tensile moduli of both kenaf and glass fibre laminates for 15 % and 22.5 % fibre volume fractions. This holds true for 30% fibre volume fraction as well, but to a lesser degree for glass fibre laminates.

The specific flexural strength is displayed in Fig. 11. It can be seen that for each of the three fibre volume fractions, the glass fibre laminates are superior to the kenaf fibre laminates. In addition, the specific flexural strength is hardly affected by the resin system at 22.5 % and 30 % fibre volume fractions for both kenaf and glass fibre laminates. The specific flexural modulus, shown in Fig. 12, exhibits a trend similar to the flexural strength with glass fibre laminates displaying better properties than those made with kenaf.

Fig. 13 presents the results for specific impact energy. The glass fibre laminates are clearly superior to the kenaf fibre laminates by about an order of magnitude. The specific impact strength also shows similar trends with the glass fibre laminates dominating the results. These results are, however, not shown here for brevity.

4.5 Water absorption tests

The increase in mass of laminates immersed in water for a period of four weeks is shown in Fig. 14. It can be seen that the kenaf fibre reinforced laminates, for any fibre volume fraction, absorb significantly more water than those made with the glass fibre. Additionally, as seen by Rouison et al. [31] for hemp fibre reinforced polyester, the mass increase of the kenaf fibre reinforced laminates increases with increasing fibre volume fraction in contrast to that of the glass fibre reinforced laminates. Furthermore, it is evident that among the kenaf fibre laminates, the ones made with vinyl ester resin absorb the least amount of water. Fig. 15 shows the increase in thickness of kenaf fibre

laminates after four weeks. It is apparent that the increase in thickness is proportional to that of mass. The increase in length and width is shown in Fig. 16. It is evident that the fibres swell more in the thickness direction of the laminate than along the length or width. This observation is expected for two reasons. Firstly, the laminate has a quasi-two-dimensional structure and so the fibres are preferentially aligned in the plane which tends to inhibit moisture induced strains in this direction. In addition, Almgren et al. [32] have observed that the hygroscopic expansion of fibres in the radial direction is larger than along their length. This effect tends to promote out of plane strains due to the in plane alignment of the fibres.

5 Conclusions

A comparison of the mechanical and water absorption properties of laminates made of kenaf fibre and three different resins, namely polyester, vinyl ester and epoxy, has been made. Addition of kenaf fibres to these resin systems results in lower tensile strengths but higher tensile moduli than those of the unreinforced resin laminates. The three different resins affect the flexural strength differently depending on the fibre volume fraction. Flexural moduli are approximately equal for all three resin systems at each of the three fibre volume fractions considered. Polyester laminates have the highest impact energy and strength, followed by the vinyl ester and then the epoxy laminates. All the above mechanical properties increase with increasing fibre volume fraction for all three resin systems.

SEM examinations of impact test specimens show that epoxy laminates failed by fibre fracture, polyester laminates by fibre pull-out and vinyl ester ones by a combination of fibre pull-out and fibre fracture. This indicates that epoxy resin bonds better to the kenaf fibres than vinyl ester or polyester resins.

The specific tensile and flexural properties for all kenaf fibre laminates are inferior to glass equivalents. At low to moderate fibre volume fractions, some of the specific properties of kenaf laminates are comparable with those of glass laminates. However, specific impact energy and strength of kenaf laminates are an order of magnitude lower than their glass equivalents for all fibre volume fractions.

All kenaf laminates absorb substantially more water than those made with glass fibres. The average value of the coefficient of hygroscopic expansion of the kenaf laminates in the thickness direction is substantially larger than those along the length or width.

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Fig. 1 Tensile strength as a function of fibre volume fraction and resin system

Fig. 2 Tensile modulus as a function of fibre volume fraction and resin system

Fig. 3 Flexural strength as a function of fibre volume fraction and resin system

Fig. 4 SEM micrographs of laminates of 15% fibre volume fraction. Voids in the vinyl ester and epoxy laminates are highlighted.

Fig. 5 Flexural modulus as a function of fibre volume fraction and resin system

Fig. 6 Impact Energy as a function of fibre volume fraction and resin system

Fig. 7 SEM micrographs of impact failure surfaces of composites for 22.5 % fibre volume fraction

Fig. 8 Impact strength as a function of fibre volume fraction and resin system

Fig. 9 Specific tensile strength as a function of fibre volume fraction and resin system

Fig. 10 Specific tensile modulus as a function of fibre volume fraction and resin system

Fig. 11 Specific flexural strength as a function of fibre volume fraction and resin system

Fig. 12 Specific flexural modulus as a function of fibre volume fraction and resin system

Fig. 13 Specific impact energy as a function of fibre volume fraction and resin system

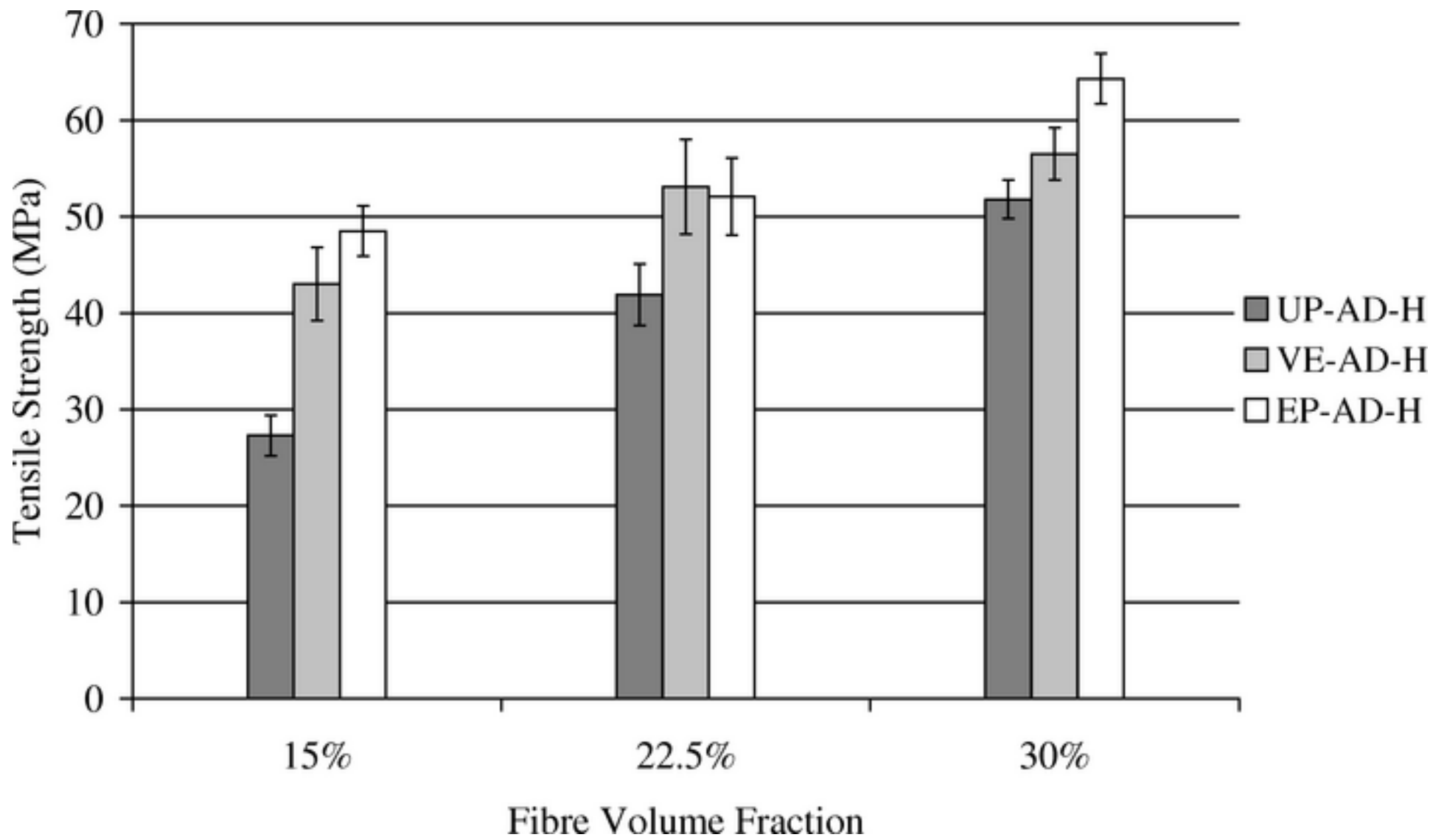
Fig. 14 Mass increase after 4 weeks of water absorption

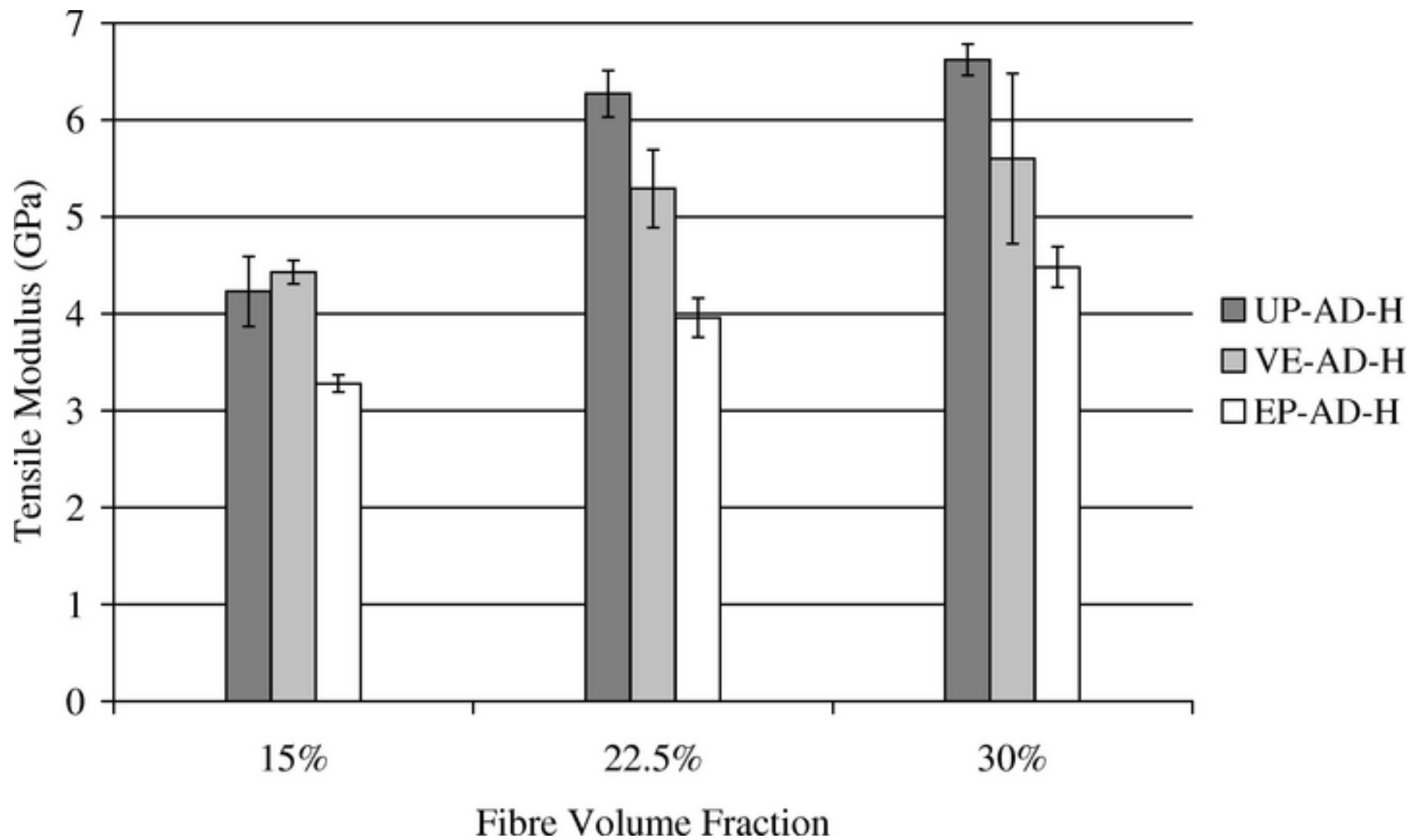
Fig. 15 Thickness increase after 4 weeks of water absorption

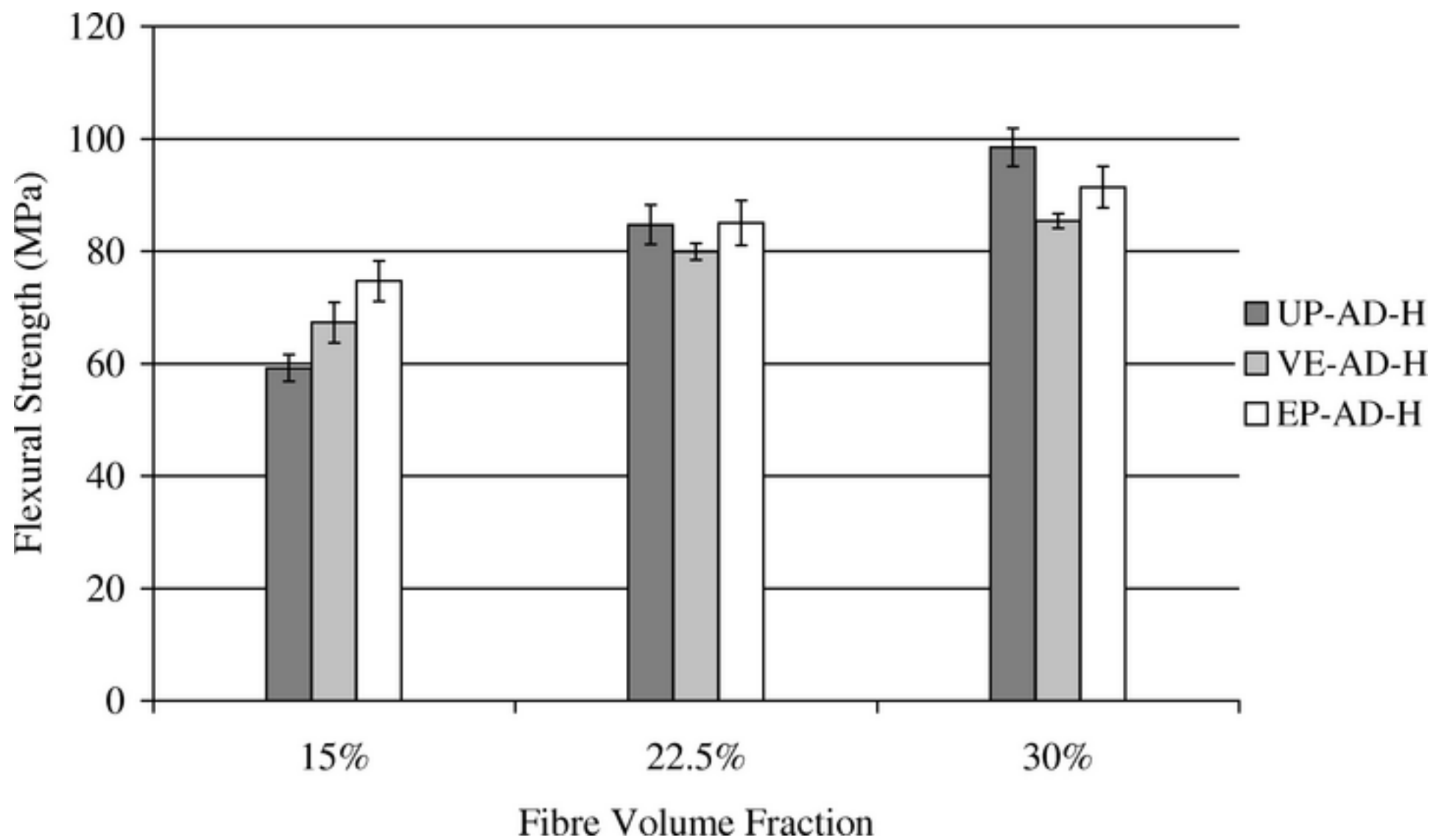
Fig. 16 In-plane dimensional increase after 4 weeks of water absorption

Table 1 Properties of kenaf fibre and various resins

Property	Unit	Kenaf Fibre	Polyester	Vinyl Ester	Epoxy
Tensile Strength	MPa	350-600 ^[19]	69 ^[20]	86 ^[21]	73.3 ^[22]
Elastic Modulus	MPa	40000 ^[19]	3800 ^[20]	3200 ^[21]	3470 ^[22]
Elongation at Break	%	2.5-3.5 ^[19]	2.3 ^[20]	5-6 ^[21]	4.5 ^[22]
Density	kg/m ³	1500 ^[19]	1140 ^[20]	1140 ^[21]	1139 ^[22]

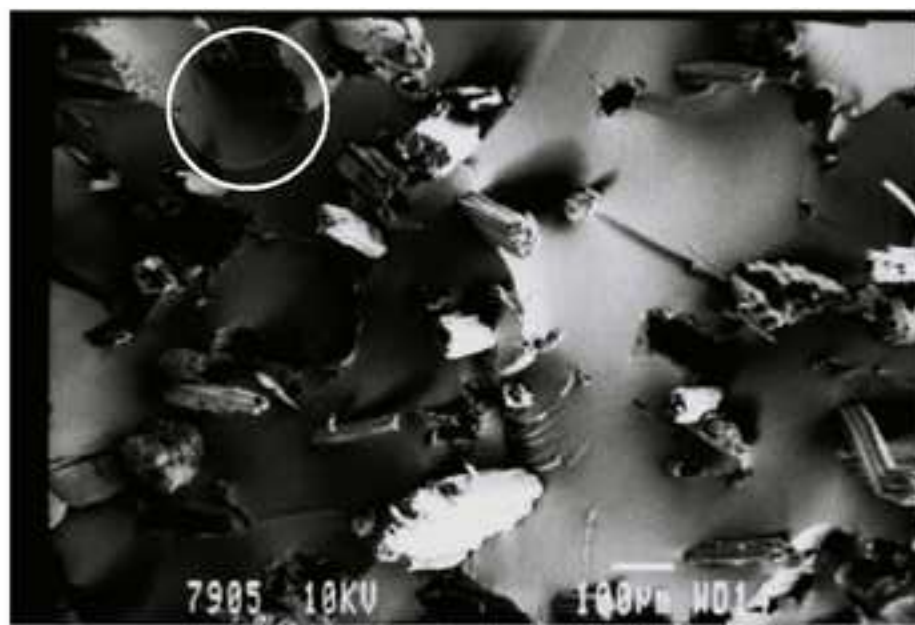




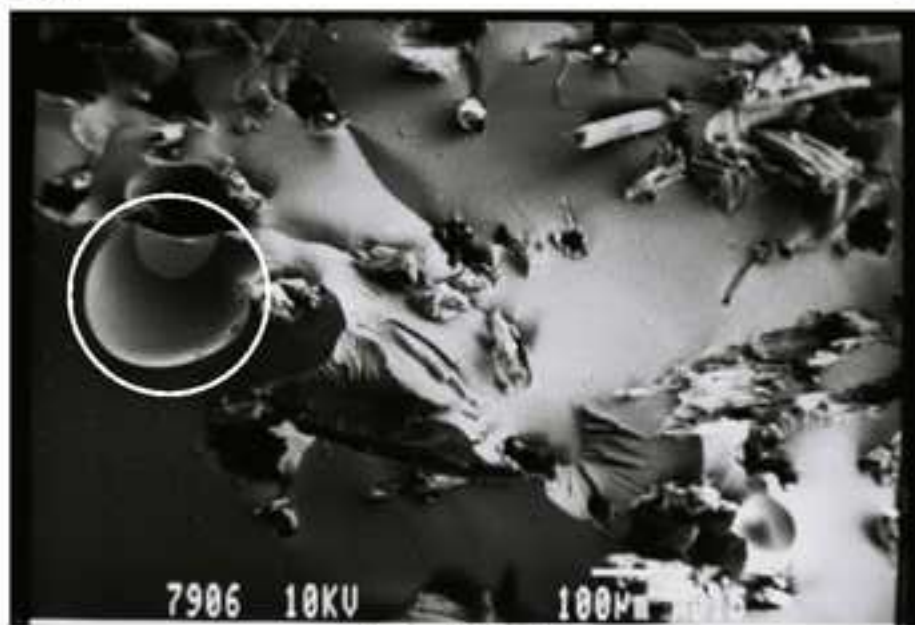




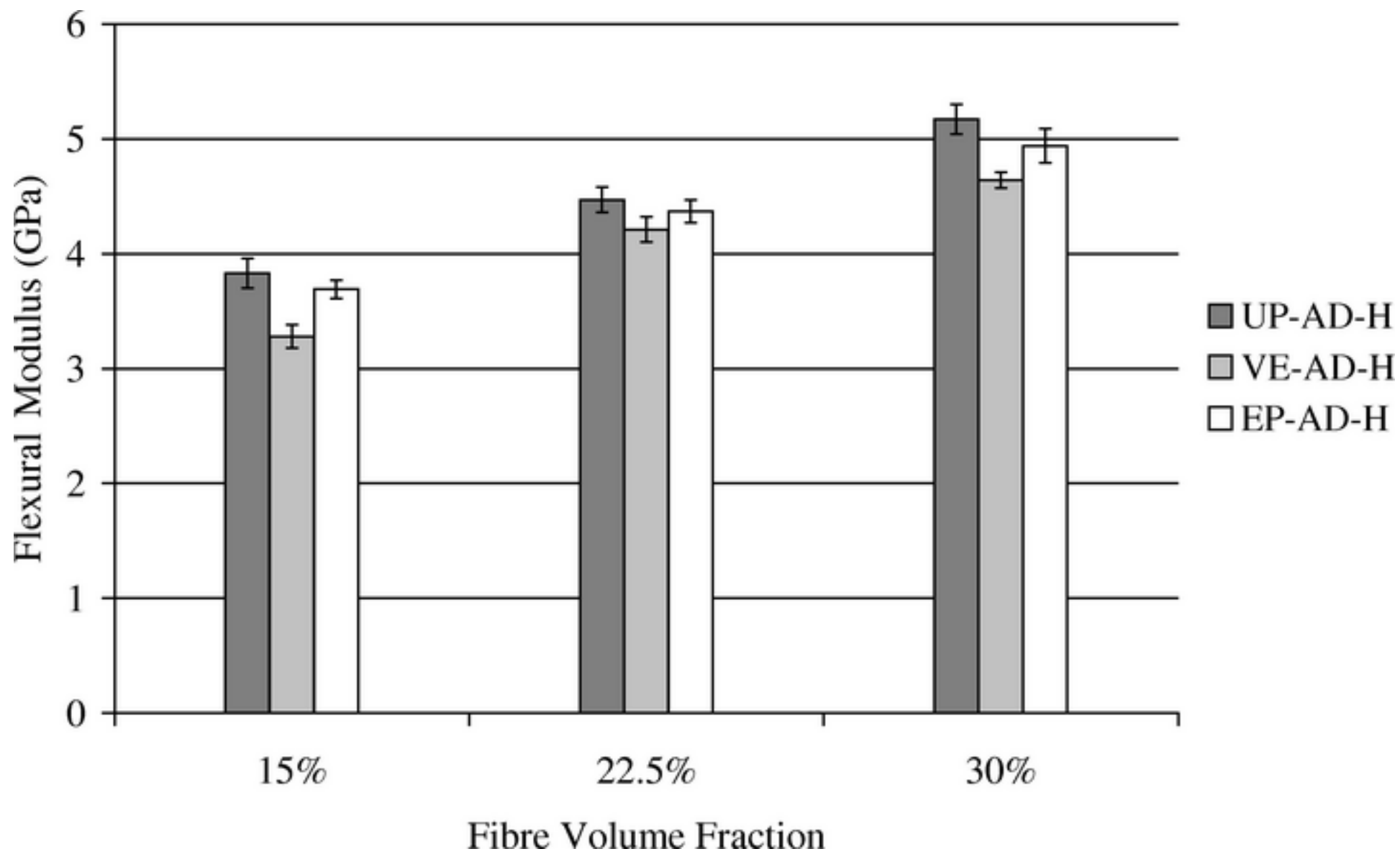
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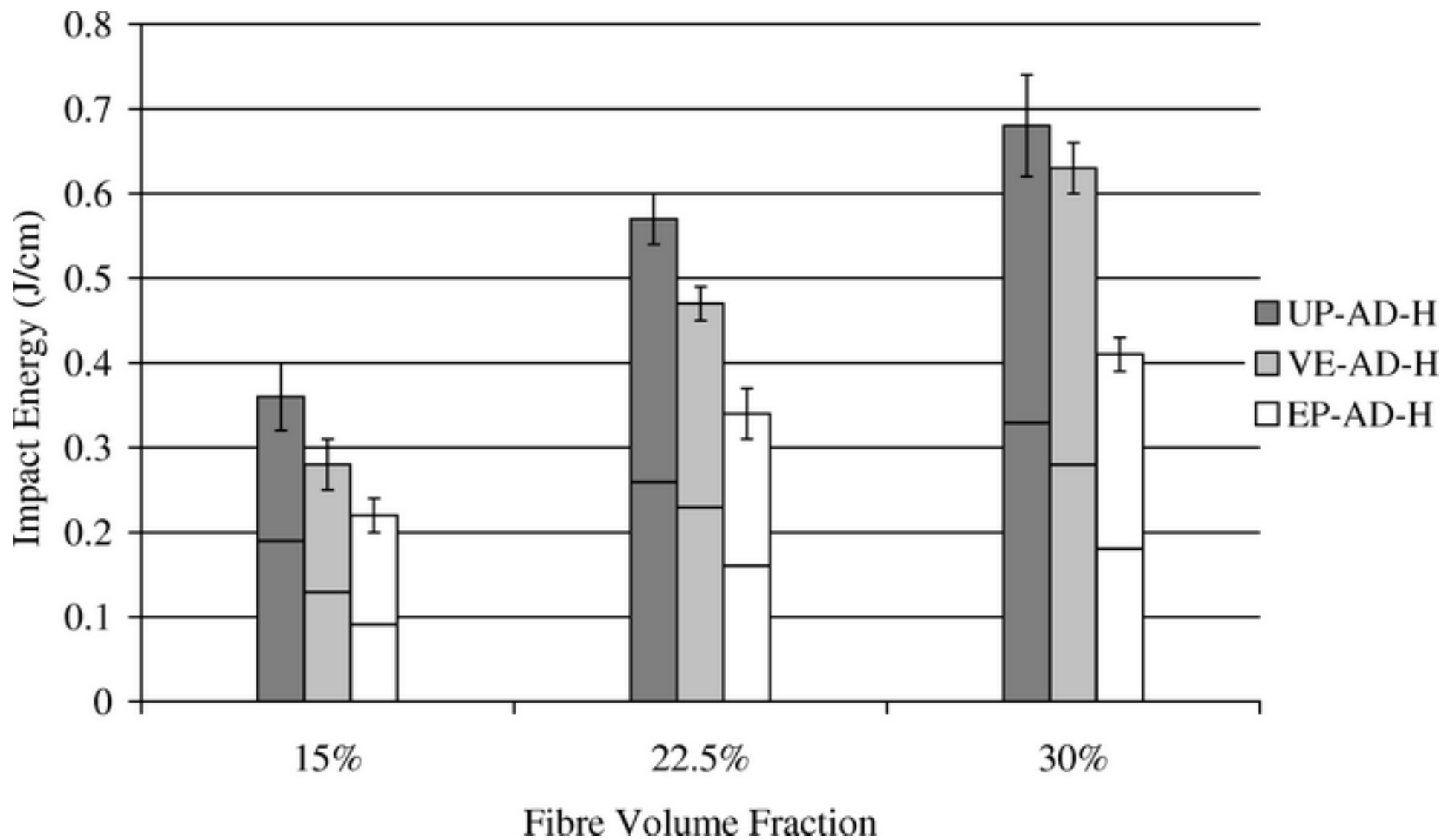


VE-AD-H



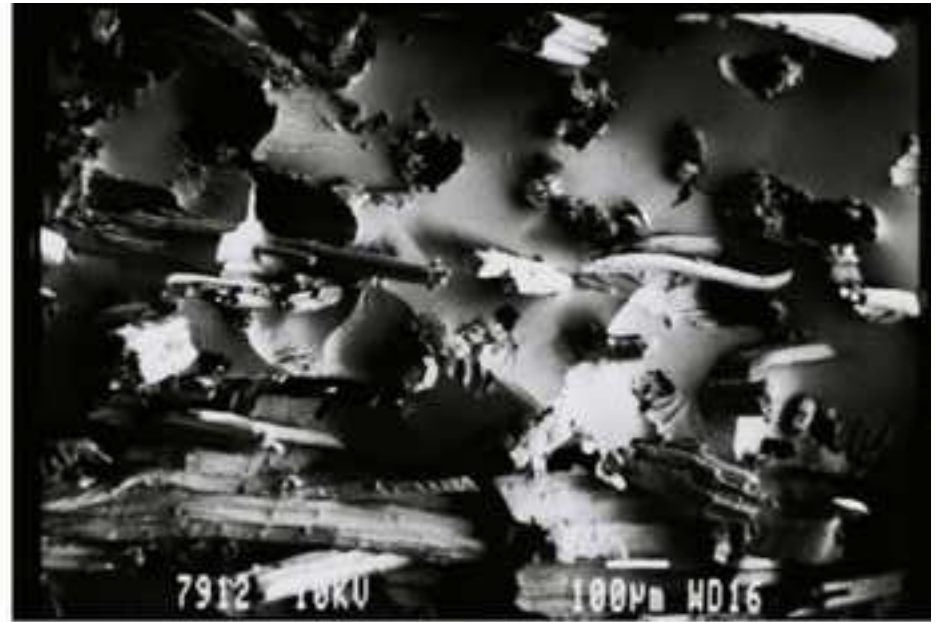
EP-AD-H



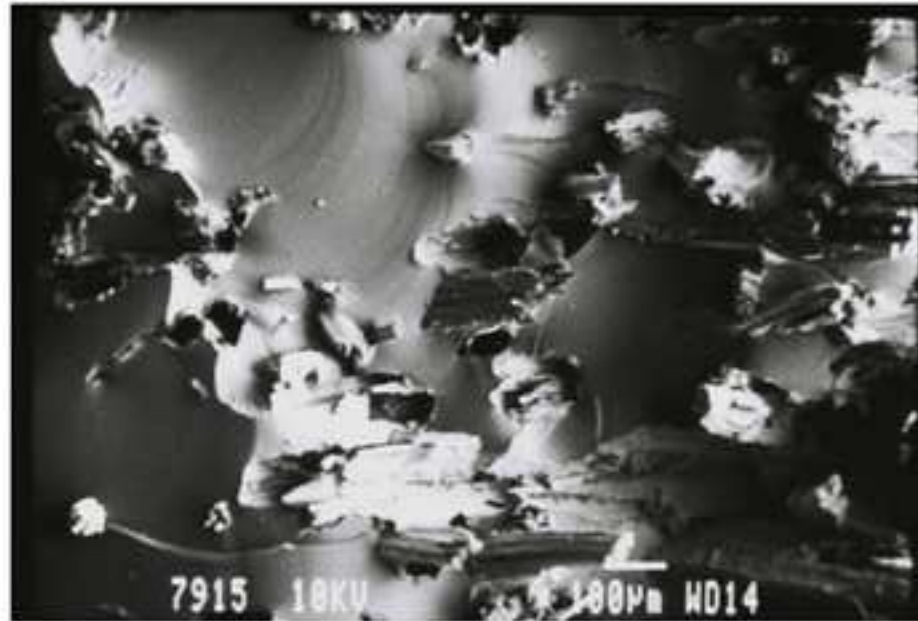




UP-AD-H



VE-AD-H



EP-AD-H

