Variability in the elastic and time-delayed properties of structural hemp fibre composites

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Abstract

Various papers in the open source literature have shown that the tensile properties of plant fibres are very scattered. To date, this difference has been viewed as an obstacle to the use of biobased composites in structural applications. However, this question of dispersion has yet not been extensively addressed at the composite scale. Therefore, this paper proposes a method to quantify the distribution of values of the monotonic and time-delayed properties of a woven hemp fabric-reinforced GreenPoxy composite. The variability in these properties is significantly lower than at the fibre scale and on the same order of magnitude as glass fibre composites. This paper also provides the suitable probability distribution laws for these properties, which are important inputs to ensure the optimized and reliable design of plant fibre composite structures.

Keywords: A. Plant fibre composite, B. Mechanical properties, B. Creep, C. Scattering

1. INTRODUCTION

The very large amount of work conducted over the last fifteen years on plant fibres has shown that their tensile properties are very scattered [1-3]. The main results available to date on this topic were recently and comprehensively reviewed by Baley et al. [4]. Even if the variability in plant fibres is comparable to glass fibres for the ultimate properties [5], the variability in stiffness properties is generally higher. This variability is attributed to the fibre extraction processes, the conditions used to prepare and characterize the fibres, and agronomic factors [4]. These last steps directly drive the main features of the fibre and its wall. In the literature, the variability in the morphology of the fibre, the ultrastructure and the constituting wall material was considered, and the effects of these parameters on the tensile behaviour of the fibres has been assessed using a sensitivity analysis [6]. The variability in plant fibres has generally been projected at the scale of composites, leading to a discourse scaring end-users and thus slowing the introduction of these fibres for structural composite applications. Indeed, this variability, if transferred to the composite's scale, might lead to an oversizing of parts, limiting the added value of plant fibres in the process of lightening structures. The effect of fibre variability on short fibre composites has been studied [7, 8]. For the design of secondary structures for marine applications, Blanchard & Sobey [9] performed a reliability analysis and showed that flax composite solutions must be 2.4 times heavier than E-glass structures to have an equivalent safety or probability of failure. However, these results were obtained using ranges and variations of mechanical properties of flax laminates collected from an extensive literature review including composite materials manufactured using various flax reinforcement patterns, manufacturing processes and fibre volume fractions. Researchers have argued that the range of properties might be significantly reduced, and then the resulting design for a particular safety factor is decreased when considering a flax material produced using a specific and mastered production process. Therefore, at this stage of development of biobased composites, a complete understanding and quantification the propagation of this variability across scales is necessary to produce rich and reliable experimental datasets allowing access to the coefficients of variation and the description of the distribution laws of the properties of biobased composites. Blanchard et al. [10] proposed an interesting multiscale characterization of the tensile properties of a woven flax epoxy composite. They compared the variability in tensile properties measured at the yarn, fabric and laminate scales, and they showed a significant decrease at the laminate scale, with variability comparable to synthetic-based composites. The coefficients of variation (CoV) of the tensile strength and rigidity of the laminates were 7.9 and 5.1%, respectively, whereas those at the yarn scales were equal to 20.3 and 18.6%. Del Masto [11] also investigated the propagation of the variability in tensile properties across scales, from the constituents of the fibre wall to the ply, for a unidirectional plant fibre epoxy composite using a stochastic multiscale model-based approach. She predicted a coefficient of variation less than 15% at the laminate scale for a coefficient of variation of the tensile properties of the constituting fibres of approximately 40 to 50%.

The uncertainties that exist in the material properties must not only be quantified but also be described by probability distributions to optimize the reliability-based design of structures and pieces fabricated from biobased composites. These data are clearly missing in the open source literature. Recent studies have also noted the significant time-delayed behaviour of plant fibre composites [12] and the need to consider it to predict long-term behaviour [13].

Therefore, the objective of the present study is to quantify the uncertainties in the elastic and time-delayed properties of structural plant fibre composites and to provide the associated probability distribution laws. The work is focused on a woven hemp fabric reinforced epoxy composite. This material was developed recently from low-twisted rovings [14] and is considered a suitable solution for semi structural applications. Therefore, the use of hemp may supplement the materials used in plant fibre reinforcement with good properties, such as the flax materials that are already marketed.

2. MATERIALS AND METHODS

2.1. Materials

The hemp reinforcement used in this study is a 6-stage hemp twill woven fabric with an areal weight of 473 g/m² [14]. The warp and weft densities were 6.5 and 9.5 yarns/cm, respectively. The twist level of the roving was equal to 40 t/m (turns per metre). For comparison, a unidirectional flax tape with an areal weight of 110 g/m² (Flaxtape L-110), provided by Ecotechnilin, was also used. This flax tape is considered the front-runner among the available marketed plant fibre reinforcements [15, 16]. With both reinforcements, an epoxy system composed of GreenPoxy 56 resin and the SD 7561 hardener, provided by Sicomin, was used. The manufacturing procedure and the thermocompression cycle of the composites were identical to those described in a previous study [12]. For both composites, a curing temperature of 60 °C for 1 hour was chosen. A postcuring step at 130 °C for 1 hour was then applied. The flax and hemp fibre-reinforced composites were labelled FUD and HWF, respectively. Tensile specimens were cut in the manufactured plates using a Trotec Speedy 300 machine along the fibre direction for the FUD composite and the weft direction for the HWF composite. The dimensions of the FUD and HWF specimens were 200×15×3.3 mm^3 and 190×15×2.2 mm^3, respectively, and the fibre volume fractions were 0.44 and 0.52 for the FUD and HWF composites, respectively. The samples were stored at 23 °C and 50% relative humidity in a Memmert HPP108L climatic chamber for at least 20 days to ensure that they reached the equilibrated moisture content.

2.2. Mechanical testing

Two different types of mechanical tests were performed on batches of 30 specimens each. The first was a monotonic tensile test up to failure. It was applied to both the FUD and HWF composites. The tests were performed using an MTS Criterion 45 machine. The strain was measured using an Instron® 2620–601 contact extensometer with a gauge length of 50 mm and a measuring range of ±10%. The crosshead speed was fixed at 1 $mm.s^{-1}$ in accordance with the ASTM D 3039 standard. Two moduli, labelled E_L^1 and E_L^2, the ultimate stress and strain were computed from the tensile curves. The moduli were determined using a linear regression analysis in the ranges between 0.01% and 0.15% strain and from 0.4% strain until failure. The mechanical setup is presented in Figure 1.

The second mechanical test was a tensile creep/recovery test. It was only performed on the HWF composite. The tests were performed on the same machine and using the same extensometer. The load rate was equal to 28 MPa s^{-1}. The unloading rate was equal to the loading rate. The durations of the creep and recovery stages were both 1 hour. The viscoelastic properties were identified using an anisotropic viscoelastic model with the inverse method from the recovery function. All the details of the testing and identification methods are also described in a previous study [12].

The scattering in the measured properties was compared to data from the literature for hemp and flax fibres [5, 17] and a glass fibre composite [9].

2.3. Statistical analysis

The adequacy of the distribution of mechanical parameter values with usual distribution laws was evaluated using Extremes software to use the data from this study in a reliability design tool [18]. Normal, lognormal, gamma, Weibull and uniform distribution laws were considered. The Anderson–Darling test statistic, labelled X, was calculated. For a specified dataset and distribution, the better the distribution fits the data, the smaller this statistic will be. The adequation of the usual distribution laws with the experimental dataset was also evaluated graphically by plotting the cumulative probability functions. The experimental frequency was calculated using the empirical frequency of Hazen, which is defined as follows:

$$\frac{i-0.5}{N} \tag{1}$$

where i is the rank in the dataset ordered by increasing values and N is the number of samples.

3. RESULTS AND DISCUSSION

3.1. Scattering of quasi-static tensile properties

The tensile curves recorded for the batches of 30 specimens of FUD and HWF composites are displayed in Figure 2 a and Figure 2 b, respectively. FUD composites present the well-known biphasic behaviour already observed and documented in the literature [19]. The monotonic tensile behaviour of the HWF composites is marked by a more pronounced nonlinearity, as already observed for composite reinforced with woven flax fabrics [20], and with a shape similar to that observed for elasto-plastic materials. The mean values, the standard deviations and the coefficients of variation (CoV) of the mechanical properties, which are defined as the ratio between the standard deviation and the mean value, are presented in Table 1. The CoVs of the modulus (E_L^1), ultimate strain and strength are equal to 8, 6.0 and 5.9% for the FUD composite and to 5.9, 8.1 and 7.5% for the HWF composite, respectively. Therefore, the scattering in the tensile properties of the woven hemp composite is of the same order of magnitude as that of the flax composite. The values measured for flax composites are also similar to those reported by Blanchard et al. [10] for a woven flax reinforced epoxy composite. The CoVs of the elastic modulus E_L^1 and the ultimate stress and strain of FUD composites were also compared to the values obtained for elementary flax fibres and glass fibre reinforced epoxy composites (see Figure 3). Compared to elementary fibres, the results highlight a scale effect characterized by scattering approximately three times lower at the scale of the composites than at the fibre scale. This decrease in variability might be explained by the averaging effect observed in the composite. Indeed, in a composite, a large quantity of fibres is tested simultaneously. This reduction is also consistent with numerical results; Del Masto *et al.* [11] predicted a CoV less than 15% for a laminate reinforced with fibres whose CoV of their tensile properties is approximately 40 to 50%. The measured properties are also subjected to a higher degree of uncertainty at the fibre scale than at the composite scale. For single plant fibres, the accuracy of the measurement is highly dependent on the fibre preparation (extraction and handling), the experimental settings (fibre alignment and clamping, gauge length, strain rate, and hygrothermal conditioning), data collection (measurement of load and displacement or strain) and postprocessing (machine compliance correction, determination of stress and modulus, loading history, assumptions related to isotropy and homogeneity).

Moreover, the scattering of the modulus and ultimate stress of the flax and hemp fibre-reinforced composites is comparable to that measured for glass/epoxy composites. The parameters of the distribution laws identified from the fitting of the experimental dataset collected for hemp-based composite are available in Table 2. A graph superimposing the experimental dataset and the fitted functions is proposed in Figure 4 for the elastic modulus E_L^1, the ultimate stress and the ultimate strain. For the modulus, the Anderson–Darling statistic test of the functions is low, indicating that they describe the experimental distribution of the elastic modulus fairly accurately, except for the Weibull and uniform laws. Regarding the strength and the ultimate strain, the test statistic is the lowest for the Weibull law, which provides the best fit of the experimental data. Conversely, the uniform law is not suitable for the description of the distribution of these parameters, as observed graphically and attested by the very high value of the Anderson–Darling test statistic.

3.2 Scattering of the creep/recovery and viscoelastic behaviour

The scatter of the time-delayed properties of the HWF composite was also studied using a batch of 30 specimens subjected to a creep/recovery test at a nominal stress of 55 MPa. The evolution of the longitudinal strain over time is displayed in Figure 3 c. The mean values, the standard deviation and the CoV of the identified time-delayed parameters are summarized in Table 1. The residual strain and the strain rate are the two most dispersed features, with CoV values reaching approximately 18% and 11%, respectively. The dispersion of the other identified parameters never exceeds 10%. The CoV values of the creep properties obtained at the composite scale were compared to those measured at the fibre scale for hemp [17] (see Figure 5). For tensile properties, the scatter of the creep properties at the composite scale is significantly lower than that at the fibre scale, with more than 10 times lower values for CoV. The values of the Anderson–Darling's statistical test were also calculated to compare the capability of the distribution laws to fit the experimental datasets. The parameters identified for the tested laws are listed in Table 2. These functions and parameters might be used advantageously in the future for the reliable design of plant fibre composite structures.

CONCLUSIONS

As the dispersion of the mechanical properties of plant fibres is viewed as a hindrance to the use of biobased composites, the variability of the monotonic and creep/recovery properties of a newly developed woven hemp fabric reinforced epoxy composite was studied and quantified in this paper. The results were compared to values measured for a unidirectional flax fibre reinforced epoxy composite, to data from the literature for woven flax fabric reinforced epoxy and glass fibre composites and for plant fibres themselves. The work highlights the following conclusions:

- The dispersion of the monotonic and creep/recovery properties is significantly lower at the composite scale than at the fibre scale. In particular, the scatter of monotonic parameters of the tested plant fibre composites is on the same order of magnitude as that of the unidirectional glass fibre composite.
- The scatter in the values of modulus, ultimate strain and strength of UD flax and woven hemp fabric reinforced epoxy composites is of the same order of magnitude.
- The identified distribution functions provided here can be used in the future for the stochastic modelling of plant fibre composites and the reliable design of structures. The calculation of the Anderson–Darling test statistic enables the identification of the distribution law that provides the best fit.

In future studies, the quantification of the effect of hygrothermal conditions on the dispersion of monotonic and creep properties will be interesting.

Acknowledgements

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Figure 1. Picture of a FUD sample before the tensile test

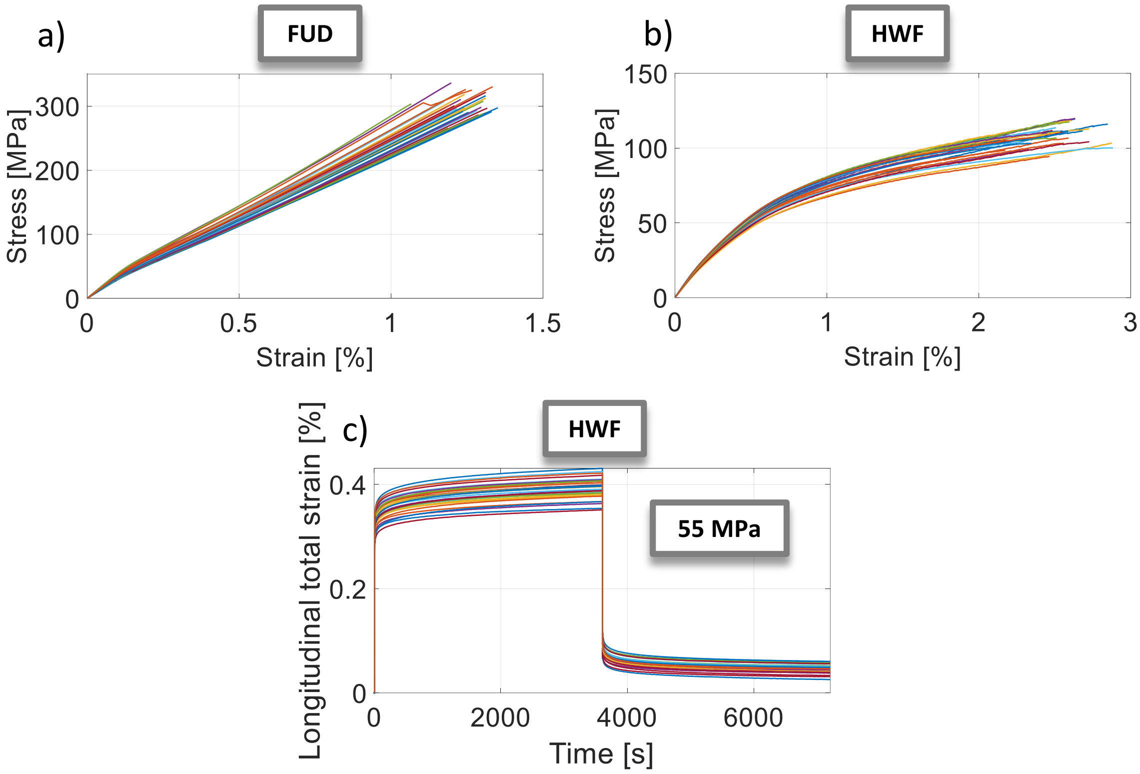

Figure 2: Quasi-static tensile test curves recorded on two batches of 30 FUD a) and HWF b) composite specimens and creep/recovery curves recorded on a batch of 30 samples of HWF composites loaded at 55 MPa.

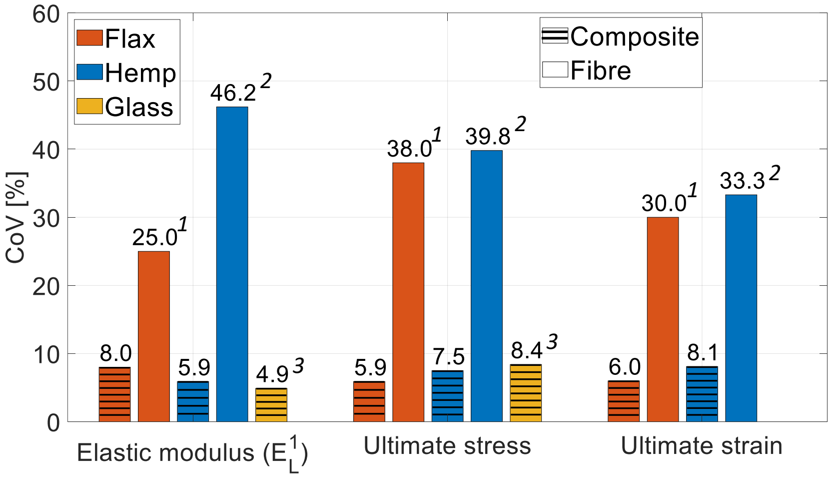

Figure 3: Coefficient of variation of the elastic properties measured on 30 different FUD and HWF composite specimens. Comparison of the value obtained to the ones published in literature for flax fibres ([1] Lefeuvre et al. [5]) and hemp fibre (Placet et al.[21]) and glass/epoxy composites ([3] Blanchard & Sobey [9]).

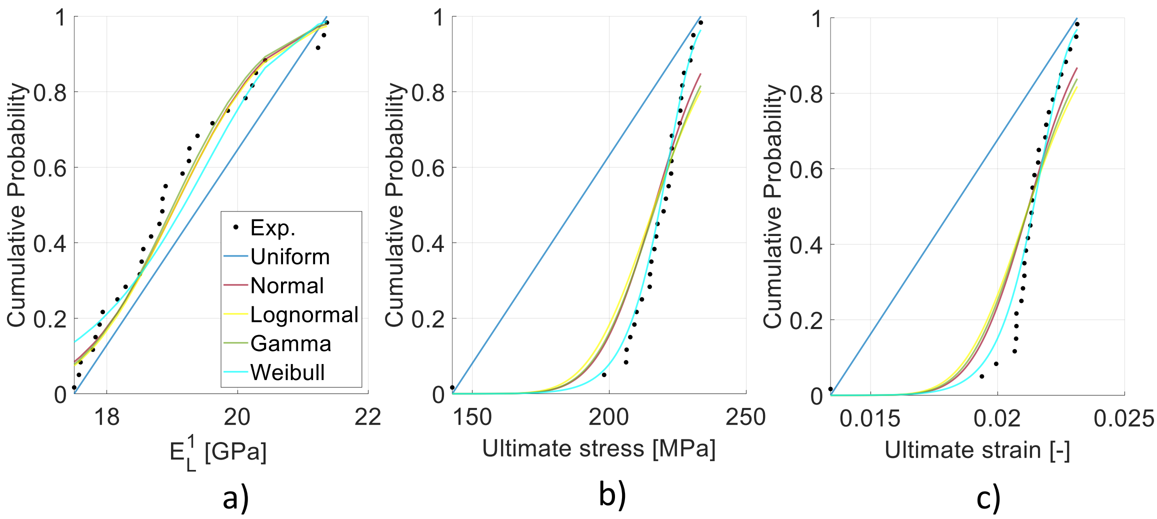

Figure 4. Plot of the experimental dataset and of the cumulative probability functions fitted using usual distribution laws in the case of modulus E_L^1 a), ultimate stress b) and ultimate strain c) for hemp fibre composite.

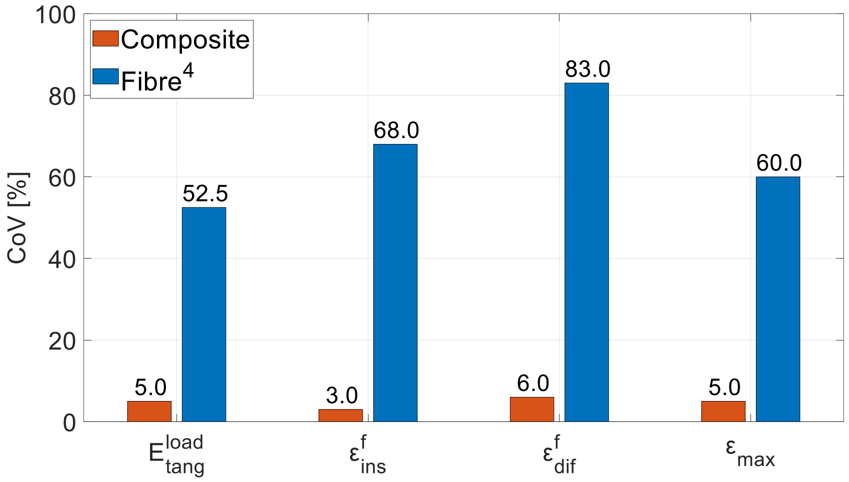

Figure 5. Comparison of the CoV of creep parameters with values from literature at hemp fibre scale ([4] Cisse et al. [17])

Table 1. Mean values, standard-deviation and coefficient of variation of quasi-static and creep/recovery mechanical properties of FUD and HWF composites

	FUD			HWF		
Mechanical properties	Mean value	Standard-Deviation	CoV [%]	Mean value	Standard-Deviation	CoV [%]
E_L^1 [GPa]	31	2	8	19	1	5.9
E_L^2 [GPa]	24	2	7.2	-	-	-
Ultimate stress [MPa]	301	18	5.9	217	16	7.5
Ultimate strain [%]	1.25	0.08	6.0	2.12	0.17	8.1
$\dot{\varepsilon}_{creep}$ [10^{-8} s^{-1}]	-	-	-	5.56	0.61	11
E_{tang}^{load} [MPa]	-	-	-	20	1	5
ε_{ins}^{c}[%]	-	-	-	0.31	0.01	3
ε_{ins}^{r}[%]	-	-	-	0.30	0.01	3
ε_{del}^{c} [%]	-	-	-	0.082	0.005	6
ε_{del}^{r} [%]	-	-	-	0.052	0.005	10
ε_{max} [%]	-	-	-	0.39	0.02	5
ε_{res} [%]	-	-	-	0.045	0.008	18
$ln(\tau_1)$	-	-	-	5.5	0.23	4.1
SD	-	-	-	3.1	0.16	5.0
β_L	-	-	-	0.23	0.02	8.6

Table 2. Values of Anderson-Darling statistic test X and parameters of the distribution laws of elastic and time-delayed properties of the hemp-based composite identified from experimental datasets.

Distrib. Law *Probability density function*	E_L^1 [MPa]	Ultimate stress [MPa]	Ultimate strain [mm/mm]	$ln(\tau_1)$	SD	β_L
Normal $f(x) = \frac{1}{\sigma_d\sqrt{2\pi}} e^{-\frac{1}{2}\left(\frac{x-m}{\sigma_d}\right)^2}$	**X=0.52** m=19058 σ_d=1133	**X=2.32** m=217 σ_d=16	**X=2.54** m=2.1.10^{-2} σ_d=1.7.10^{-3}	**X=1.27** m=5.50 σ_d=0.23	**X=0.99** m=3.10 σ_d=0.16	**X=0.24** m=0.23 σ_d=0.02
Lognormal $f(x) = \frac{1}{x\sigma_d\sqrt{2\pi}} e^{-\frac{1}{2}\left(\frac{\ln(x)-m}{\sigma_{dl}}\right)^2}$	**X=0.44** m=9.85 σ_d=0.059	**X=3.13** m=5.4 σ_d=0.088	**X=3.46** m= -3.86 σ_d=0.095	**X=1.11** m=1.7 σ_d=0.04	**X=0.87** m=1.13 σ_d=0.05	**X=0.24** m=-1.47 σ_d=0.09
Gamma $f(x) = \frac{x^{p-1}e^{-\frac{x}{\theta}}}{\Gamma(p)\theta^p}$	**X=0.47** p=298 θ=63.9	**X=2.72** p=150 θ=1.45	**X=3.00** p=129 θ=1.65.10^{-4}	**X=1.09** p=608 θ=0.009	**X=0.87** p=377 θ=0.008	**X=0.25** p=125 θ=0.002
Weibull $f(x) = \frac{\beta}{\eta}\left(\frac{x}{\eta}\right)^{\beta-1} e^{-\left(\frac{x}{\eta}\right)^\beta}$	**X=1.02** η=19605 β=16.9	**X=0.51** η=222 β=24.0	**X=0.88** η=2.18.10^{-2} β=21.0	**X=8.39** η=5.62 β=18.2	**X=4.60** η=3.18 β=17.3	**X=0.38** η=0.24 β=12.4
Uniform $f(x) = \frac{1}{b-a}$	**X=1.77** a=17367 b=21500	**X=17.80** a=140 b=237	**X=16.75** a=1.3.10^{-2} b=2.3.10^{-2}	**X=6.87** a=4.99 b=6.40	**X=3.02** a=2.78 b=3.54	**X=0.75** a=0.19 b=0.27