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An Alternative Way of Measuring Tensile Characteristics of Handsheets

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Abstract − In this work, the laboratory-made handsheets having different grammages were obtained at certain production conditions. The tensile characteristics of the laboratory-made handsheets were analysed by using two measuring techniques. The main method for determining tensile behaviour was performed on the universal testing machine, which is a generally accepted conventional testing machine. A rheometer device was used as a second and alternative method for measuring the tensile properties of handsheets. The given data were investigated and compared with the data of conventional tensile tester. Linear least square regression was applied to analyse the association of the data from the Instron 5564 Universal Testing Machine and Hybrid Rheometer Discovery HR-2 Machine with examined tensile characteristics depending on the various grammage range. The experiments on the handsheets having different grammage groups showed that the methods give results in reasonable agreement. These results showed that there are acceptable differences in the tensile index and elastic modulus values of the samples as a function of their basis grammages.

Keywords − *Handsheets, tensile characteristics, tensile index, rheometer, universal testing machine*

Laboratuvar Yapımı Kağıtların Çekme Özelliklerini Ölçmenin Alternatif Bir Yolu

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Araştırma Makalesi

Öz − Bu çalışmada, belirli üretim koşullarında farklı gramajlara sahip laboratuvar yapımı el yaprakları elde edilmiştir. Laboratuvarda üretilen el yapraklarının çekme özellikleri iki ölçüm tekniği kullanılarak analiz edilmiştir. Çekme davranışını belirlemek için ana yöntem, genel olarak kabul edilen geleneksel bir test makinesi olan evrensel test makinesinde gerçekleştirildi. El yapraklarının çekme özelliklerini ölçmek için ikinci ve alternatif bir yöntem olarak bir reometre cihazı kullanıldı. Reometre cihazından alınan veriler incelendi ve geleneksel çekme test cihazı verileriyle karşılaştırıldı. Instron 5564 Universal Test Cihazı ve Hibrit Discovery HR-2 Reometre Cihaızndan alınan verilerin çeşitli gramaj aralığına bağlı olarak incelenen çekme özellikleriyle ilişkisini analiz etmek için doğrusal en küçük kareler regresyonu uygulandı. Farklı gramaj gruplarına sahip kağıtlar üzerinde yapılan deneyler, yöntemlerin makul bir uyum içinde sonuçlar verdiğini göstermiştir. Bu sonuçlar, numunelerin temel gramajlarının bir fonksiyonu olarak çekme indeksi ve elastik modül değerlerinde kabul edilebilir farklılıklar olduğunu göstermiştir.

Anahtar Kelimeler − *Laboratuvar yapımı kağıtlar, çekme özellikleri, çekme indeksi, reometre, universal test cihazı*

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1. Introduction

Testing of raw materials, finished products and intermediates in manufacturing processes is a common feature of all industrial processes. While a product is being manufactured, or after manufactured is qualified with several forms of testing. Efficient and relevant testing becomes more important as industrial processes become more sophisticated and tolerances for fluctuations in product characteristics become more stringent. All test methods attempt to numerically describe a particular relevant characteristic or characteristic of a product (Gullichsen et al. 1999). These tests measure parameters that correlate well with the properties of the product in question. It is important to evaluate the results of paper tests to determine the suitability of the paper material for the area of application and to check whether it meets the expected product requirements (Mark 2002).

Determining the strength of a paper is an intensive research topic in paper physics. The strength changes of paper property were examined by the studies of Page (Page 1969) and Seth (Seth 1995). Tensile testing has become a fundamental strength test for understanding the structural dynamics of paper materials. The tensile test is used to find out how strong a material is and also, how much it can be stretched before it breaks. In the literature, different attempts have been made to describe the behaviour of paper under stress using experimental, semi-empirical and computer modelling methods (Cox 1952; Page 1969; Seth 1995; Van den Akker 1962; Perkins and Mark, 1976; Nilsen et al. 1998; Heyden 2000; Miettinen et al. 2007). Also, the influence of paper grammage on its tensile strength has been investigated by many researchers (Burgess 1970; Mohlin 1992; Retulainen and Nieminen 1996; Winters et al. 2002; Gülsoy et al. 2016; Gülsoy and Şimsir 2017)

The purpose of this study is to show that the rheometer device is an alternative measurement technique that can be safely used in the absence of access to a universal testing machine for the tensile testing of paper materials. Evaluation of the results of tests performed on both devices using the same group of samples proves the applicability and reliability of the force-elongation curve obtained with a rheometer to determine the tensile properties of the paper material.

In this work, the load and extension curves of handsheets made in laboratory conditions were measured with a Universal tensile tester device and a rheometer device. Essentially, a laboratory rheometer can be used to measure how a liquid and suspension flows in response to an applied force. It cannot be determined from a single viscosity value, so it is used for liquids that need to set and measure more parameters than is the case with a viscometer. The goal is to measure the paper material with a rheometer device and compare the obtained data with the data from a Universal test device. Hence, the question of whether the rheometer device could be used as an alternative device in paper material tensile testing was the subject of this work.

2. Materials and Methods

2.1. Raw Material and Pulp Evaluation

Spruce softwood bleached kraft pulp was used in papermaking. The bleached pulp was initially soaked in 5 liters of distilled water for at least 4 hours to prepare it for beating. The beating time was applied for 200 minutes using a Valley Beater following ISO 5264-1:2019. Before making laboratory-made handsheets, the pulp was fractionated using a Somerville fractionator (Tappi T 275 - 2018) equipped with an 80 mesh screen. This procedure was used to control the length of the fibres and to exclude fine particles from the pulp furnish. Before and after the fractionation steps, the drainage resistance of the selected pulp and the amount of water retained in the wet pulp mass was characterized by Schopper-Riegler values according to ISO 52671: 2021 and water retention values according to Tappi UM 256 (2015). The morphological characteristics of the above-mentioned cellulose fibers without fine particles were determined with a Metso FS5 fiber analyzer.

2.2. Handsheet Making Process

The principles of TAPPI T 205 (2018) were applied in the formation procedure. The handsheets were formed with predetermined grammages, namely 10, 20, 30, 40, 50 g m $^{-2}$. The required amount of pulp for the target grammages was calculated precisely and determined systematically in accordance with another related work (Engin 2017). After the forming procedure, a laboratory type hydraulic press was operated and the pressure was gradually increased for formed handsheets webs according to the standards. The pressed handsheets were dried at room conditions (22° C~ and 65% relative humidity) for 48 hours.

2.3. Physical Testing of Handsheets

The samples were conditioned according to the ISO 187:1990 standard before the physical and mechanical tests. An electronic micrometer was used to determine the sample thickness according to the ISO 534: 2011 standard. Five layers of laboratory-made handsheet samples were stacked and the measurements at various points were statistically evaluated. These thickness measurements were determined in terms of a micron (µm). Each handsheet was produced as having a 200 cm^2 surface area. The grammages of the handsheets were given as the unit of g•m-^{2} and these were determined regarding the ISO 536:2012 standard with a balance sensitive to 0.001 g. When determining the physical properties of the handsheets, the grammage groups of the specimens were examined separately and evaluated in this way. The density of handsheets was specified as the units of g•cm-^{3} and these were calculated as in conformity with the Standard ISO 534:2017.

2.4. Mechanical Testing of Handsheets

Mechanical testing was performed on two different devices; Hybrid Rheometer Discovery HR-2 Machine and an Instron 5564 Universal Testing Machine (Figure 1). Sample sizes were prepared in two different sizes. These were defined as 15 cm x 1.5 cm for the Universal device and 4 cm x 0.4 cm for the rheometer device, respectively.

a **b**

Figure 1. The devices used for tensile testing; a) the Instron 5564 Universal Testing Machine and b) the Hybrid Rheometer Discovery HR-2 Machine.

The ISO 19242: 2018 standard was taken into account in the calculation of the tensile index value. In addition, each sample was weighed individually to calculate confidence intervals for all samples (I`Anson and Sampson, 2007). The elastic modulus was estimated from the slope of the steepest part of the load-extension curves.

The tensile strength, the tensile index (specific tensile strength), the elastic modulus and the specific elastic modulus of specimens were calculated by the following Equations (2.1-2.5), where TS is tensile strength (N m^{-1}), F is breaking load (N), wsp is specimen width (m), T is the tensile index (N m g^{-1}), βsp is specimen grammage (g m $^{-2}$), SMax is the maximum slope of the curve (Nm $^{-1}$), ΔF is the force increment (N), Δε is the elongation increment (m), E is the elastic modulus of paper (N m^{-1}), lsp is specimen width (m) and Es is the specific elastic modulus of paper (Nm g^{-1}).

$$TS = \frac{F}{\mathrm{wsp}} \tag{2.1}$$

$$T = \frac{TS}{\beta\mathrm{sp}} \tag{2.2}$$

$$SMax = (\frac{\Delta F}{\Delta\varepsilon})Max \tag{2.3}$$

$$E = \frac{SMax * lsp}{\mathrm{wsp}} \tag{2.4}$$

$$Es = \frac{E}{\beta\mathrm{sp}} \tag{2.5}$$

The Least-squares regression was used to analyze tensile index data from two measures where the sequential grammages of handsheets were consistently correlated.

3. Results and Discussion

3.1. Fiber and Pulp Characteristics

The information of the quantity of retained water within the beaten and fractionated fines-free pulps and their water retention values are given in Table 1.

Table 1
The values of Schopper-Riegler (°SR) and water retention (WRV) for Whole pulp(WP) and fines-free (FF) pulp

Beating Time (min)	°SR		WRV (g/g)	
	Whole Pulp	**Fines-free Pulp**	**Whole Pulp**	**Fines-free Pulp**
200	54	17	3.43	3.15

The changes in the values of Schopper-Riegler (°SR) and water retention value (WRV) of pulps have been seen by the fractionation of the beaten pulp. These differences have been occurred due to the existence of fine fibres (Ferreira et al. 2000; Hubbe and Heitmann, 2007; Guo et al. 2009; Winter et al. 2021) and their tendency of water absorption. The morphological characteristics of the pulp used to make the sheets are shown in Table 2.

Table 2
The morphological parameters of fines-free pulp

Beating Time (min)	Fiber Length, λ (mm)	Fiber Width, ω (µm)	Fiber Coarseness, δ (mg/m)
200	2.39	21.6	0.153

Fiber length is considered an important variable among fiber parameters. The effect of fibre length has been discussed previously (Clark, 1962; Page, 1969; Wangaard and Woodson, 1972; Seth, 1995; Nordström, 2014). It is known that the increase in fiber length positively affects the tensile strength of the paper (Page 1969; Nordström 2006; Nordström 2014). The variation in the fibre length affects paper formation, fibre-fibre bonding mechanisms and consequently the strength efficiency between fibres (Nordström 2006). For this reason, the length-frequency distribution of fibres was taken into account. In this experimental work, metıculously fractionated fibres were used as pulp furnish. The length-frequency distribution of utilized fibre furnish is presented in Figure 2. The average fibre length was measured around 2.4 mm. The frequency distribution of fibres having 2.5 mm length is more distinct as seen in Figure 2.

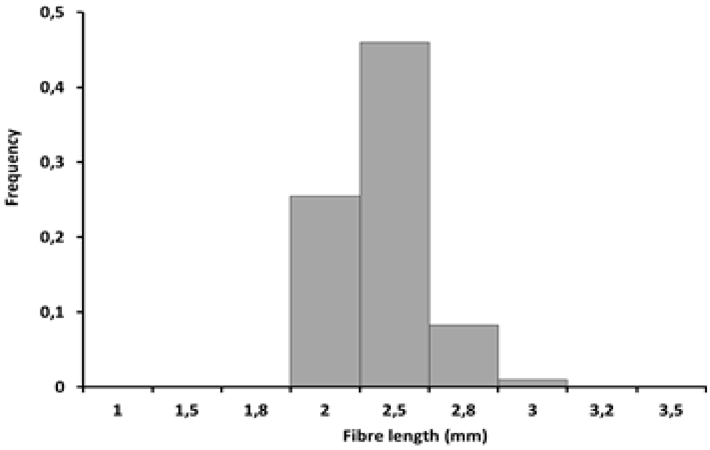

Figure 2 The fibre length frequencies for the fines-free pulp

3.2. Handsheet Characteristics

The paper density value of a paper determines its structure and hence its mechanical characteristics (Ingmanson and Thode, 1959). In Figure 3, the density values of handsheets are shown according to considered grammages.

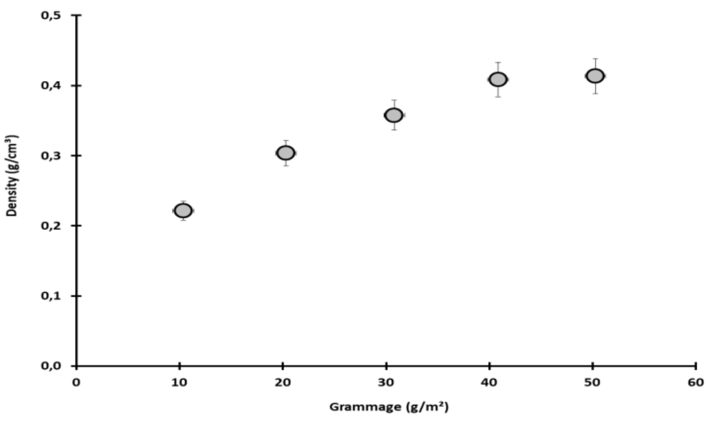

Figure 3 Density values of laboratory-made handsheets

The increase in the paper grammage has been caused that the paper density values for given handsheets have increased. This is because the increase in paper size and thickness is not a constant ratio. The paper is a porous material. During the paper forming section, the fibres settle down in the available spaces in the fibre network and its surface layers. The thickness of a paper shows a nonlinear increase as the grammage of paper (Bloch et al. 2019). Concordantly, the fundamental characteristics of a paper such as structural and mechanical properties are affected due to this change in density values (Ingmanson and Thode, 1959; I'Anson et al. 2008).

The measurement of tensile strength is expressed with the loading of a strip of a paper specimen in an axial direction until it breaks. The tensile testing machine displays load and tensile data. It also includes an omittable initial stage. At the start of a tensile test, as the jaws separate, slack in the specimen is arranged and aligned vertically before the load cell re-applies a significant load. The load and strain curves for all analyzes have been normalized and thus zero load and strain points have been determined (in Figures 4 and 5) at a load of 0.01 Fmax.

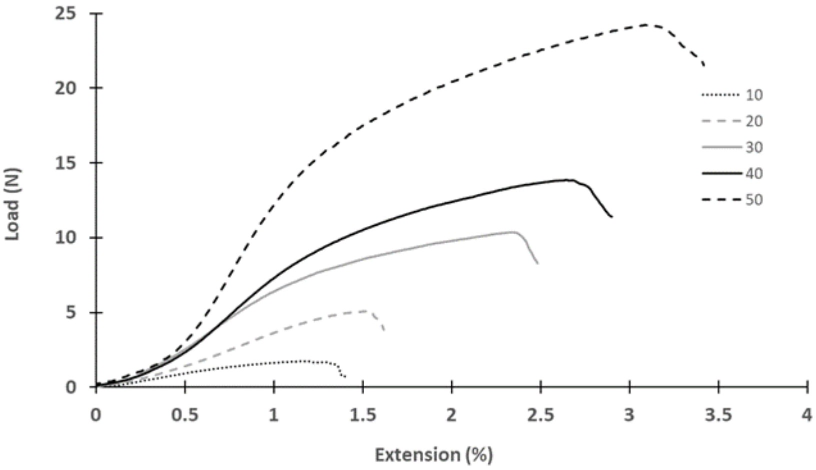

Figure 4 Load and extension curves plotted from the data of the Instron 5564 Universal Testing Machine

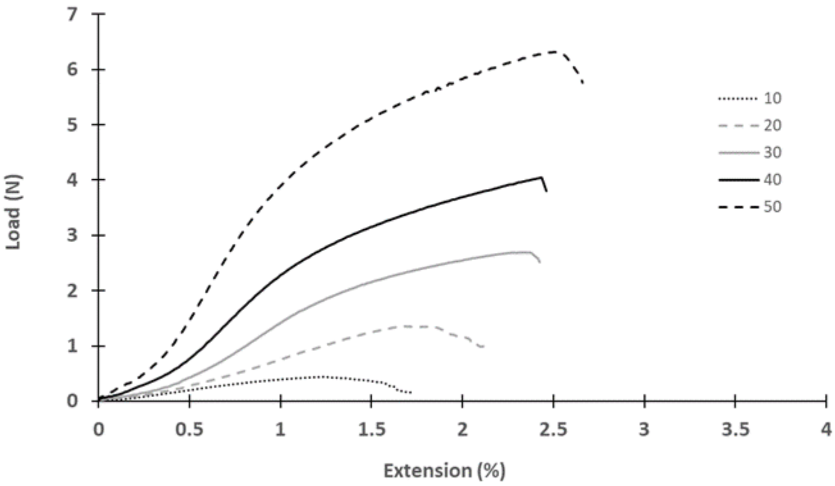

Figure 5 Load and extension curves plotted from the data of the Hybrid Rheometer Discovery HR-2 Machine

The calculated tensile index and specific elastic modulus values of the handsheets are plotted as bar charts in Figures 6 and 7. The tensile index and specific elastic modulus values are influenced by the interactions of fibres in the paper network. This issue is closely related to stress-transfer efficiency among fibres, as explained previously (I'Anson and Sampson, 2007). The findings in Figures 6 and 7 can mainly have been characterised the mechanical behaviour of paper and also exhibit a dependence on grammage, they increase with grammage. In this experimental work, the increasing tendency of tensile data is seen as a continuous logarithmic function, but indeed this increasing tendency is limited (I`Anson and Sampson, 2007). Similarly, other previous studies have shown that tensile strength values are affected by tests performed on different grammages and paper types (Retulainen and Nieminen 1996; Winters et al. 2002; Gülsoy et al. 2016; Gülsoy and Şimsir. 2017). However, rather than understanding the overall trend of tensile characteristics of paper, this study has been performed only on limited grammage groups to confirm the coherence of the two measurement methods.

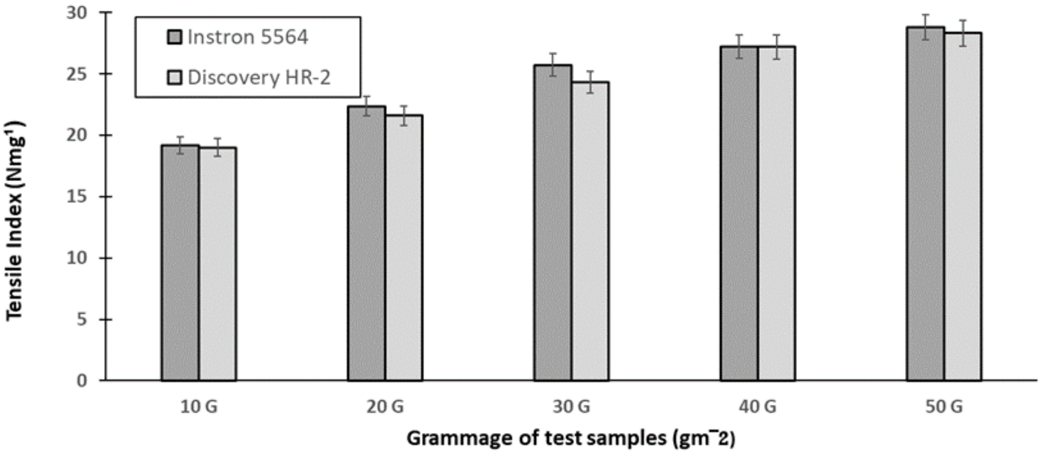

Figure 6 Comparison of tensile index values of paper groups obtained from two measurement ways

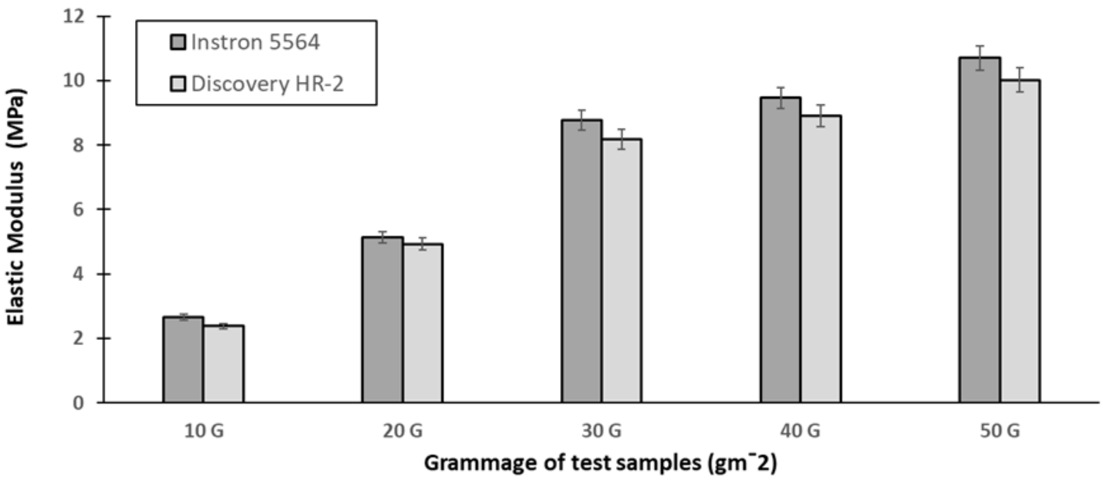

Figure 7 Comparison of elastic modulus values of papers in given different grammages obtained from two measurement ways

Figures 6 and 7 show that the obvious differences in the bar charts are primarily due to the differences in grammages of the handsheets. In Figures 6 and 7, the error bars have been given to the standard deviation of measurements of individual handsheets at given grammages according to the defined measurement methods. The total number of fibres bonded in the thickness direction of the handsheet influences the force distribution behaviour within the network. As the number of bonded fibres increases, the layer structure becomes clearer and the tensile stress can be better transferred among fibre layers. The same Figures (Figure 6 and 7) have been also demonstrated that the differences in the values of the tensile index and elastic modulus are minimal for a given grammage of handsheets with two measurement techniques.

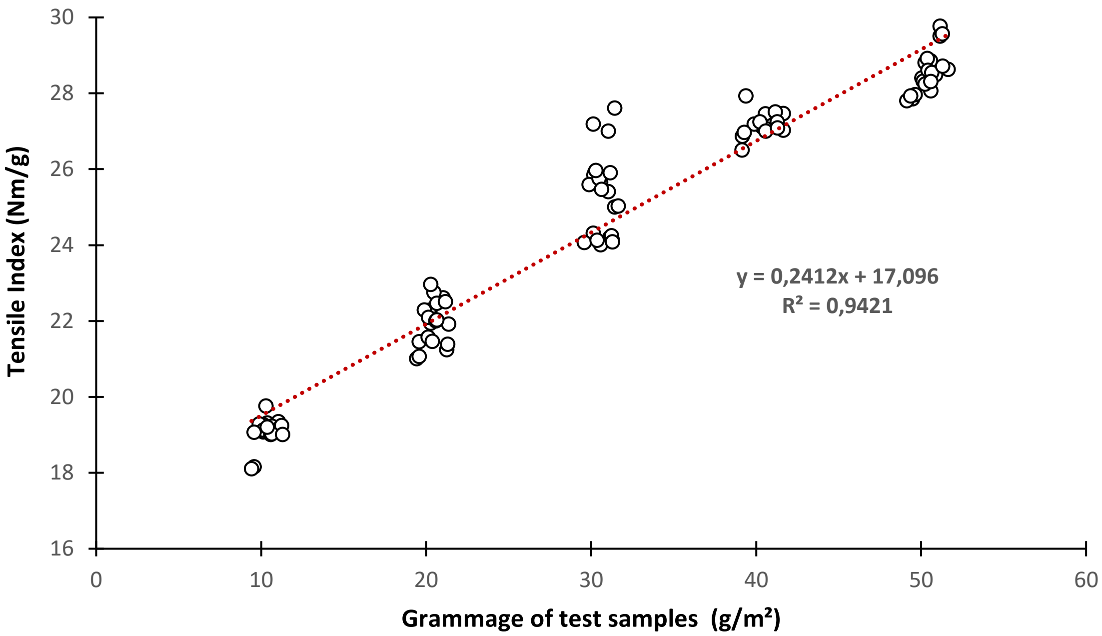

Figure 8 Applied least squares regression analysis for two measurement methods

The least-squares regression method has been employed for the analysis of the data of two measurement methods in which successive grammages are serially correlated (in Figure 8). The data has been collected from the Instron 5564 Universal Testing Machine and Hybrid Rheometer Discovery HR-2 Machine as previously explained.

Results are shown for a range of tensile indices of several handsheets at a given grammage. These indicated that a good agreement has been achieved between measurements using two different devices. The gradient is 0.24 which is the coefficient of a linear regression of the data of tensile index and grammage. A large positive linear association has been seen accordingly in the equation in Figure 8. The R^2 is found as the 0.94 this means that the points are close to the linear trend line.

4. Conclusions

Paper specimens with a given grammage value were evaluated among themselves. It has been found that the tensile index and modulus values obtained by the measurements from the Rheometer Discovery HR-2 and the Tensile Instron 5564 device were very close to each other. The mean and standard deviation of the measured specimens have also been supportive of these results. The tensile characteristics of handsheets have been different due to the change in their grammages and thus their density. However, it has been confirmed that both tensile tester and rheometer are required sensitive machines for data acquisition. These particular measurement techniques can be accepted as appropriate for the measurements of tensile behaviours of all grammage groups.

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