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Mechanical Properties and Microstructural Characterization of Paper Produced from Rice Straw - Waste Paper Pulp Blends via Ethanol Organosolv Process

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Abstract
The utilization of lignocellulosic agricultural waste as an alternative raw material for paper production offers a sustainable approach to reducing dependence on wood-based pulp. This study investigated the mechanical properties and microstructural characteristics of paper produced from blends of rice straw organosolv pulp and recycled waste paper pulp at a 1:1 ratio using 15 wt% polyvinyl acetate (PVAc) as binder. Sixteen paper samples were prepared under various ethanol concentrations (15–60 wt%) and cooking times (60–120 min). Mechanical characterization included tensile strength, elongation of break, and Young's modulus, while surface morphology was analyzed using Scanning Electron Microscopy (SEM). The tensile strength ranged from 2.06 to 4.22 MPa, elongation of break from 6.00 to 16.06%, and Young's modulus from 0.155 to 0.529 MPa. All samples satisfied the minimum tensile strength requirement of SNI 14-6519-2001. SEM observations revealed that higher tensile strength was associated with denser and more homogeneous fiber networks with fewer voids and surface defects. The optimum tensile strength was achieved at 30 wt% ethanol and 60 min cooking time, indicating favorable fiber bonding and structural integrity.
Keywords: *rice straw pulp, organosolv paper, recycled paper, tensile strength, sem morphology*

Abstrak
Pemanfaatan limbah lignoselulosa pertanian sebagai bahan baku alternatif pembuatan kertas merupakan pendekatan berkelanjutan untuk mengurangi ketergantungan terhadap pulp berbasis kayu. Penelitian ini menganalisis sifat mekanik dan karakteristik mikrostruktur kertas yang dibuat dari campuran pulp organosolv jerami padi dan pulp kertas bekas daur ulang dengan rasio 1:1 menggunakan perekat polivinil asetat (PVAc) sebesar 15 wt%. Sebanyak 16 sampel kertas diproduksi dengan variasi konsentrasi etanol 15–60 wt% dan waktu pemasakan 60–120 menit. Karakterisasi mekanik meliputi kuat tarik, elongasi putus, dan Modulus Young, sedangkan morfologi permukaan dianalisis menggunakan Scanning Electron Microscopy (SEM). Hasil penelitian menunjukkan bahwa kuat tarik berkisar antara 2,06–4,22 MPa, elongasi putus 6,00–16,06%, dan Modulus Young 0,155–0,529 MPa. Seluruh sampel memenuhi standar minimum kuat tarik SNI 14-6519-2001. Hasil SEM menunjukkan bahwa kertas dengan kuat tarik lebih tinggi memiliki jaringan serat yang lebih rapat, homogen, dan minim rongga maupun cacat permukaan. Kondisi optimum diperoleh pada konsentrasi etanol 30 wt% dan waktu pemasakan 60 menit yang menghasilkan ikatan serat dan integritas struktur terbaik.
Kata Kunci: *pulp jerami padi, proses organosolv, kertas daur ulang, kuat tarik, morfologi sem*

1. Introduction

The mechanical integrity of paper is the primary determinant of its functional suitability across application domains ranging from printing and packaging to decorative and specialty uses. Key mechanical parameters tensile strength, elongation of break, and Young's modulus reflect the interplay between fiber type, fiber bonding density, pulp preparation conditions, and the nature and quantity of any adhesive binders employed [1]. Understanding how these parameters are governed by upstream processing conditions is therefore essential for rational process design in sustainable paper manufacturing.

A companion paper by the authors [2] reported the organosolv pulping of rice straw using ethanol as the cooking solvent, and characterized the effects of ethanol concentration (15–60% wt) and cooking

time (60–120 min) on pulp yield, cellulose content, and lignin content. That work established that ethanol concentration and cooking time are primary drivers of pulp chemical composition: higher concentrations and longer times increase cellulose content (maximum 62.08%) and reduce lignin content (minimum 29.45%), although lignin re-deposition was observed at extreme conditions (60% ethanol, 120 min). Preliminary tensile data for 60- and 80-minute cooking conditions were also presented, indicating values of 2.65–4.22 MPa.

This paper extends that work by presenting the complete mechanical characterization of paper produced from all 16 experimental conditions, with particular attention to the 100- and 120-minute cooking groups that were not reported previously. Scanning Electron Microscopy (SEM) analysis of selected samples provides microstructural evidence to explain the observed mechanical trends. Together, these data establish a comprehensive structure–property relationship for rice straw organosolv paper, enabling evidence-based optimization of manufacturing parameters.

Waste paper pulp (HVS type) was incorporated as a secondary fiber source at a 1:1 ratio with rice straw pulp, and polyvinyl acetate (PVAc) was used as a binder at 15% wt. This blending strategy reflects a circular economy approach, utilizing two waste streams simultaneously agricultural residue (rice straw) and post-consumer paper waste to produce a functional material with reduced environmental footprint [3, 4].

2. Material and Methods

Pulp Preparation

Rice straw pulp was produced via ethanol organosolv pulping as described in detail in the companion publication [5]. In brief: 12 g of 40-mesh rice straw powder was cooked with ethanol solutions (15, 30, 45, or 60% wt) at a 10:1 liquid-to-solid ratio, 110°C, 100 rpm, for 60, 80, 100, or 120 minutes in a sealed beaker reactor on a hot plate. After cooling, the solid pulp fraction was separated by vacuum filtration. Waste paper (HVS type, from photocopying waste) was soaked at 1:1 (w/v) in water for 120 minutes and blended into a pulp slurry..

Paper Manufacturing

For each of the 16 experimental conditions, rice straw pulp was blended with waste paper pulp at a 1:1 weight ratio. PVAc adhesive was incorporated at 15% wt of total pulp mass (1.5 g per 10 g total pulp) [6]. The mixture was poured onto a paper casting mold and oven-dried until a uniform sheet formed. Sheet dimensions were standardized to accommodate ASTM D638 Type IV test specimen geometry: total length 11.5 cm, narrow section width 0.6 cm, narrow section length 3.3 cm, total width 1.9 cm, gauge length 2.5 cm, grip distance 6.5 cm.

Tensile Testing

Tensile strength, elongation of break, and Young's modulus were measured using an Electronic Universal Testing Machine (Hung Ta HT-8503) at a crosshead speed of 10 mm/min. Specimens were gripped at an initial gauge length of 25 mm. Tensile strength was calculated as the maximum load at fracture divided by the cross-sectional area (MPa). Elongation of break was calculated as (extension at break / initial gauge length) × 100%. Young's modulus was derived as the ratio of tensile strength to elongation of break [7]. Raw data from the testing machine (in kgf/mm²) were converted to MPa using the factor 1 kgf/mm² = 9.807 MPa.

SEM Analysis

Scanning Electron Microscopy was performed using a JEOL JSM-6360LA instrument at the Materials Physics Laboratory, Universitas Syiah Kuala, and cross-referenced with data from the Geology Laboratory, Institut Teknologi Bandung. Paper samples (5 mm × 5 mm) representing the highest and lowest tensile strength conditions were mounted on bronze stubs, gold-sputter-coated, and imaged in Secondary Electron Image (SEI) mode at an accelerating voltage of 10 kV. Cross-sectional images were acquired at magnifications of 1000× and 5000×. Fiber diameter was estimated from scale bar measurements.

3. Results and Discussion

The mechanical performance of paper produced from rice straw organosolv pulp and recycled waste paper pulp was strongly influenced by ethanol concentration and cooking time during the pulping process. Variations in these parameters affected fiber delignification, cellulose exposure, and inter-fiber

bonding, which subsequently determined the tensile strength, elongation of break, and stiffness of the resulting paper. In addition, SEM observations provided microstructural evidence regarding fiber distribution, surface morphology, and void formation that explain the differences in mechanical behavior among samples. The complete experimental results are discussed in detail in the following sections.

Complete Mechanical Properties Dataset

The full mechanical properties dataset for all 16 experimental conditions is presented in **Table 1**. Raw tensile values (kgf/mm²) from the testing machine and derived MPa values, elongation of break (%), and Young's modulus (MPa) are reported. This complete dataset spans all four cooking time groups and enables a systematic analysis of both concentration and time effects on paper mechanical performance.

Table 1. Complete Mechanical Properties of Paper Across All 16 Experimental Conditions

Cooking Time (min)	Ethanol (%wt)	Tensile Strength (kgf/mm²)	Tensile Strength (MPa)	Elongation (%)	Young's Modulus (MPa)
60	15	0.27	2.648	13.72	0.193
60	30	0.43	4.217	16.06	0.263
60	45	0.32	3.138	8.56	0.367
60	60	0.40	3.924	9.24	0.424
80	15	0.41	4.021	7.88	0.510
80	30	0.31	3.040	8.40	0.362
80	45	0.38	3.727	9.44	0.395
80	60	0.40	3.923	8.04	0.488
100	15	0.24	2.354	7.16	0.329
100	30	0.22	2.158	13.92	0.155
100	45	0.37	3.629	8.92	0.407
100	60	0.38	3.727	8.20	0.455
120	15	0.35	3.432	11.04	0.311
120	30	0.21	2.059	6.00	0.343
120	45	0.41	4.021	7.60	0.529
120	60	0.31	3.040	15.04	0.202

All 16 samples exceeded the SNI 14-6519-2001 minimum tensile strength standard of 1.63 kN/m for base paper, validating the overall approach of using rice straw organosolv pulp blended with waste paper pulp as a functional papermaking system [8].

Effect of Ethanol Concentration on Tensile Strength

The relationship between ethanol concentration and tensile strength is non-monotonic and depends strongly on cooking time group, as shown in **Fig. 1** and summarized in **Table 2**. At 60 minutes, the highest tensile strength (4.22 MPa) was observed at 30% ethanol rather than at higher concentrations. At 80 minutes, the maximum (4.02 MPa) shifted to 15% ethanol. At 100 minutes, the peak was at 45% ethanol (3.63 MPa), while at 120 minutes the maximum (4.02 MPa) again appeared at 45% ethanol.

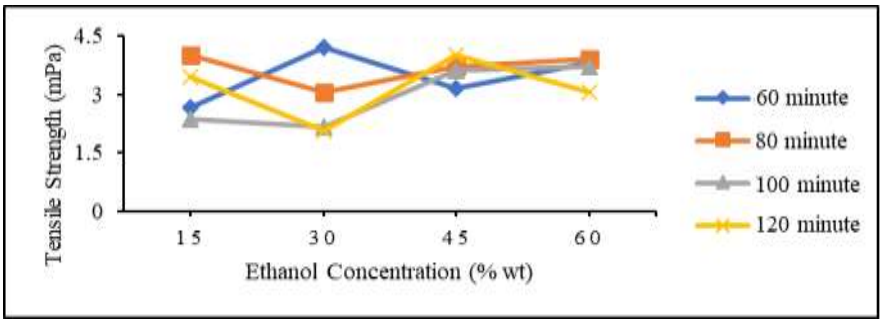

Fig. 1: Effect of Ethanol Concentration on Tensile Strength

This non-linear behavior reflects a competition between two opposing effects of delignification on paper strength. As ethanol concentration increases, more lignin is removed, which: (a) increases cellulose content and inter-fiber hydroxyl bonding capacity—strengthening the paper; but simultaneously (b) removes residual lignin that contributes to matrix stiffness and inter-fiber adhesion in non-bleached papers [9-11]. The optimum ethanol concentration for maximum tensile strength therefore represents a balance point between these effects, which shifts with cooking duration as the cumulative delignification exposure changes.

Table 2. Tensile Strength Summary by Cooking Time Group

No.	15% wt (MPa)	30% wt (MPa)	45% wt (MPa)	60% wt (MPa)	Cooking Time (min)
1	2.648	4.217	3.138	3.924	60
2	4.021	3.040	3.727	3.923	80
3	2.354	2.158	3.629	3.727	100
4	3.432	2.059	4.021	3.040	120

Elongation of Break Analysis

Elongation of break ranged from 6.00% (30% ethanol, 120 min) to 16.06% (30% ethanol, 60 min) across all conditions, as presented in **Fig. 2** and **Table 1**. In general, higher ethanol concentrations tended to reduce elongation, consistent with the hypothesis that stronger cellulose–PVAc interactions at higher cellulose content produce a stiffer, less ductile paper structure [12]. However, significant scatter was observed across conditions, notably the high elongation at 60% ethanol and 120 minutes (15.04%) which deviates from the general trend.

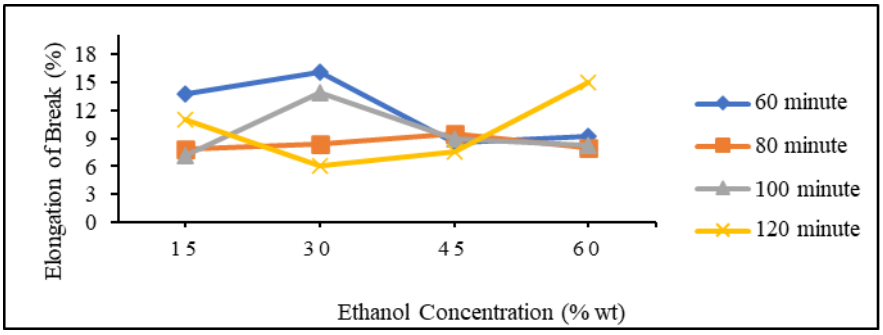

Fig. 2: Effect of Ethanol Concentration on Elongation of Break

This variability is attributed primarily to non-uniform fiber distribution during manual paper casting. In a laboratory setting, manual casting cannot guarantee consistent fiber orientation and distribution across the sheet, resulting in localized weak points that cause premature elongation before fracture. This limitation is particularly pronounced for longer-cooked pulps, which tend to produce shorter fiber fragments that are more susceptible to agglomeration during casting [13].

Young's Modulus and Stiffness Analysis

Young's modulus values ranged from 0.155 MPa (30% ethanol, 100 min) to 0.529 MPa (45% ethanol, 120 min), as shown in **Fig. 3** and **Table 1**. Modulus represents material stiffness higher values indicate papers that resist deformation under load without yielding [14]. The wide range (approximately 3.4-fold from minimum to maximum) reflects the sensitivity of paper stiffness to pulp chemical composition and inter-fiber bonding architecture.

As illustrated in **Fig. 3**, the 60-minute cooking group, Young's modulus increased progressively with ethanol concentration (0.193 MPa at 15% to 0.424 MPa at 60%), suggesting a consistent stiffening effect as cellulose purity increases. This trend was less consistent in longer cooking groups, where re-deposition phenomena and fiber degradation introduce additional variables [15, 16]. The highest stiffness at 45% ethanol and 120 minutes may reflect a fortuitous combination of moderate cellulose content and lignin re-deposition that creates a more densely cross-linked fiber network.

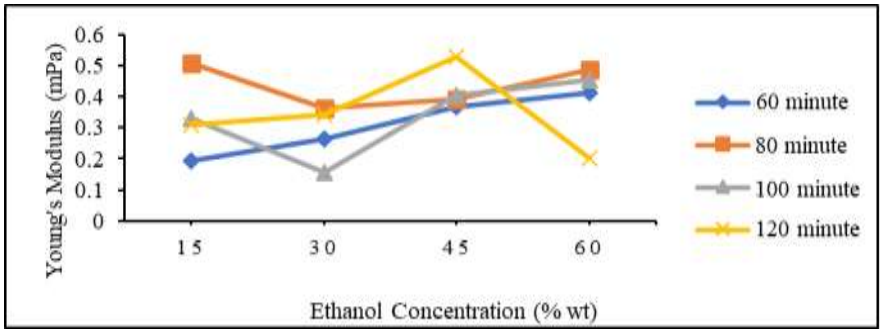

Fig. 3: Effect of Ethanol Concentration on Young's Modulus

SEM Microstructural Analysis

Scanning Electron Microscopy was performed on paper cross-sections from the highest tensile strength condition (30% ethanol, 60 min, 4.22 MPa) and the lowest tensile strength condition (30% ethanol, 120 min, 2.06 MPa) at magnifications of 1000× and 5000×. As shown in **Fig. 4**, fiber diameters observed in SEM images were approximately 10,000 nm (10 µm), consistent with reported dimensions for delignified rice straw fibers.

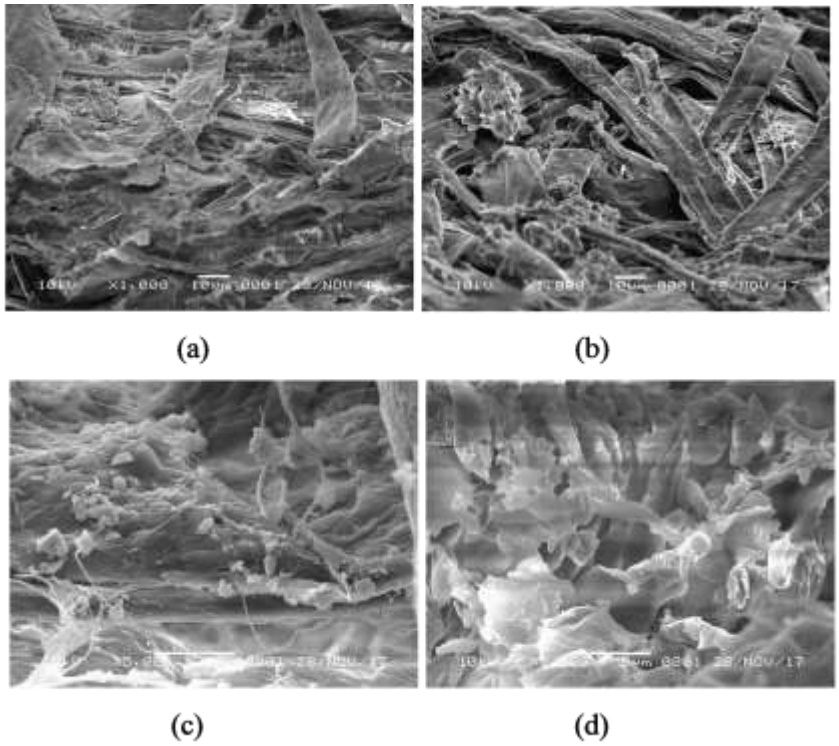

Fig. 4: SEM micrographs of paper cross-sections at different tensile strength conditions: (a) low tensile strength sample at 1000× magnification, (b) high tensile strength sample at 1000× magnification, (c) low tensile strength sample at 5000× magnification, and (d) high tensile strength sample at 5000× magnification.

The high-tensile-strength paper (30% ethanol, 60 min), presented in **Fig. 4**(b) and **Fig. 4**(d) exhibited a dense, uniform fiber network at both magnification levels. At 1000×, the cross-section showed tightly interlocked fiber bundles with minimal visible void space between fiber layers. At 5000×, individual cellulose fibers were observed to be in close contact, with PVAc adhesive visibly bridging inter-fiber junctions. The smooth, continuous surface morphology at this condition confirms effective fiber bonding, which is the microstructural basis of high tensile resistance [17, 18]. No surface cracks, delamination planes, or poly-crystalline boundary lines were visible, indicating a homogeneous material structure.

In contrast, the low-tensile-strength paper (30% ethanol, 120 min, 2.06 MPa) showed a markedly different microstructure in **Fig. 4**(a) and **Fig. 4**(c). At 1000×, the cross-section revealed rougher fiber surfaces and regions of poor fiber consolidation, with visible inter-fiber gaps. At 5000×, the fiber surface appeared irregular and partially coated with what appears to be re-deposited lignin material, consistent with the re-deposition mechanism identified from yield and chemical composition data. These surface irregularities reduce the effective contact area between fibers, impairing hydrogen bonding and PVAc adhesion, and explaining the significantly lower tensile strength [19].

Structure–Property Relationships and Practical Implications

Integrating the mechanical property data with the pulp chemical composition data from the companion study [2] yields the following structure–property relationships: (1) Higher cellulose content in the pulp correlates positively with tensile strength, up to an optimum level (approximately 54–58% cellulose), beyond which the effect plateaus or decreases due to concurrent fiber degradation. (2) Lignin content shows a U-shaped relationship with tensile strength: very high lignin (>55%) weakens paper by disrupting fiber bonding, while very low lignin (<35%) may weaken inter-fiber matrix adhesion in non-bleached papers where bleaching and surface treatments are not applied. (3) The 30% ethanol, 60-minute

condition represents the optimal balance for maximum tensile strength, while 45% ethanol, 120 minutes represents the optimum for maximum stiffness.

For practical applications, rice straw organosolv paper at the optimal conditions is suitable for use as decorative paper, book covers, tissue box covers, and other non-structural applications where a combination of adequate tensile strength (>3 MPa), moderate flexibility, and natural aesthetic is desired. The non-bleached, organosolv process aligns with clean production principles by eliminating sulfur compounds and enabling ethanol solvent recovery for reuse [20, 21].

4. Conclusion

This study successfully evaluated the mechanical properties and microstructural characteristics of paper produced from blends of rice straw organosolv pulp and recycled waste paper pulp under various ethanol concentrations and cooking times. All paper samples satisfied the minimum tensile strength requirement of SNI 14-6519-2001, demonstrating the potential of rice straw as a sustainable alternative raw material for paper production. Tensile strength, elongation of break, and Young's modulus were significantly influenced by pulping conditions, particularly ethanol concentration and cooking duration.

The optimum tensile strength (4.22 MPa) was achieved at 30% ethanol and 60 minutes, while the highest stiffness (0.529 MPa) was obtained at 45% ethanol and 120 minutes. SEM observations confirmed that superior mechanical performance was associated with denser and more homogeneous fiber networks with fewer voids and surface defects. Overall, the study demonstrates that the organosolv process combined with recycled paper pulp has strong potential for producing environmentally friendly non-wood paper with satisfactory mechanical performance.

5. Acknowledgment

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