

ADVANCES IN BIO-BASED HEAT-RESISTANT COATINGS FOR THERMALLY RESISTANT PAPER FOR ELECTRONIC CIRCUIT BOARD APPLICATIONS

THANH NGUYEN THI,* SON CAO VAN** and HANG NGUYEN THI**

**Hanoi University of Science and Technology, Vietnam*

***Research Institute of Pulp and Paper Industry, Vietnam*

✉ *Corresponding author: T. Nguyen Thi, ngthithanh1410@gmail.com*

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Cellulose-based paper is a renewable material that can replace laminated materials, such as Nomex, in electronic circuit boards, but its poor thermal stability limits its application. This study developed a technological process for producing heat-resistant paper from kraft pulp. The effects of acid purification, refining degree, and strength additive were investigated, and the paper was surface-treated with a heat-resistant polymer. Laboratory-scale production gave a paper with a basis weight of 160.6 g/m², a thickness of 0.22 ± 0.03 mm, a tensile strength of 8.31 kN/m, and a tear strength of 1955 mN. Thermogravimetric analysis showed an onset decomposition temperature of 301.8 °C, and infrared spectroscopy confirmed the presence of the heat-resistant coating with siloxane bonds. At the end of its life, the paper can be recycled into other paper products, such as industrial packaging paper. The coated paper is a promising candidate for printed circuit board applications.

Keywords: heat-resistant paper, pulp and paper, cellulose, printed circuit boards, recycling

INTRODUCTION

The rapid growth of the electronics industry has produced a large volume of electronic waste. Conventional printed circuit board (PCB) substrates, such as glass fiber-reinforced epoxy (FR-4), are petroleum-based and difficult to recycle.^{1,2} For multilayer hot-pressing, many companies use laminated Nomex sheets (aromatic polyamide with heat resistance of 180–220 °C), which are costly, imported, and not environmentally friendly.³ PCBs also account for approximately 3–17.5% of electronic waste by weight and contain heavy metals that hinder recycling.⁴

Cellulose-based paper is a renewable and biodegradable material that can replace laminated materials, such as Nomex, in electronic circuit boards, but its poor thermal stability limits its application. Heat-resistant kraft paper is therefore being considered as a lighter, renewable replacement, and the kraft paper segment of the global PCB laminate market is expected to reach USD 0.29 billion by 2028 (CAGR 8.7%). However, it must withstand hot pressing at 150–220 °C,⁵⁻⁶ while keeping good electrical insulation and strength, which is difficult for native cellulose because it is flammable and degrades above about 200 °C. Recent reviews describe biodegradable, bio-based, and paper- or cellulose-based materials as ways to reduce the environmental burden of electronics.⁷⁻¹⁰ Such a substrate must combine thermal stability and mechanical performance with a sustainable end of life. Because the paper developed here is cellulose-based and the silicone coating accounts for only about 2.5% of its mass, it can be recycled after use into other paper products, such as industrial packaging paper.

This study aimed to develop a technological process for producing heat-resistant paper from kraft pulp for electronic circuit board applications. The effects of acid purification, refining degree, and strength additive were investigated. The paper was coated with a heat-resistant polymer, and the coated paper was characterized by thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and mechanical tests. Photographs of the developed paper are presented.

EXPERIMENTAL

Materials

The chemical pulp (kappa number 24) was imported from the United States; the strength additive (starch, 99.9%) was sourced from suppliers in Vietnam; and the surface coating chemicals were provided by Wacker

Chemie AG (Germany). All chemicals were used as received, without further purification, unless specified otherwise.

The equipment used in this study included a PFI mill refiner, a PFI beater, a Rapid-Köthen sheet former, and a laboratory-scale surface sizing/coating device manufactured at the Research Institute of Pulp and Paper Industry (RIPPI).

Paper preparation process

The preparation of heat-resistant paper involved several key stages, including pulp disintegration, refining, incorporation of additives and fillers, sheet formation, surface sizing, drying, and final conditioning, as illustrated in Figure 1.

Chemical pulp was first disintegrated in a 5 L laboratory disintegrator for 10 min. The pulp was then refined using a PFI mill to achieve the desired freeness level. Subsequently, the refined pulp was blended with chemical additives under controlled conditions. The pulp suspension was thoroughly mixed and formed into laboratory sheets using a Rapid-Köthen sheet former in accordance with ISO 5269-3, with a target basis weight of 160 ± 5 g/m², followed by pressing and drying at 93 °C.

Prior to testing, all samples were conditioned under standard atmospheric conditions to ensure stability. Surface sizing was conducted using a laboratory-scale two-roll size press with an optimized formulation and processing conditions. The applied coating weight ranged from 2.0 to 4.0 g/m². After surface treatment, the samples were air-dried for 4 h and subsequently oven-dried at 93 °C. The dried samples were then conditioned under standard conditions before further evaluation of their physical and mechanical properties.

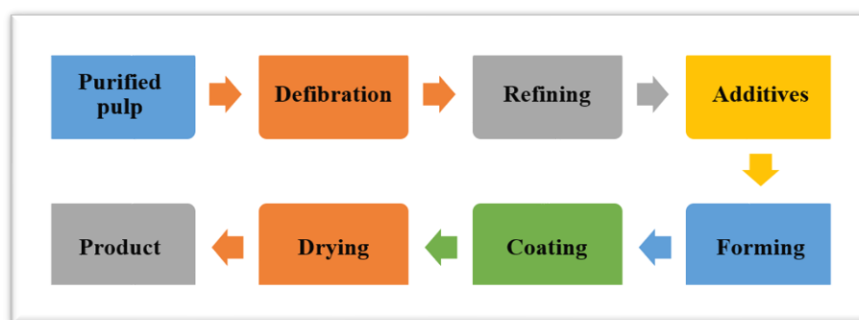


Figure 1: Block diagram of the paper manufacturing process

Characterization methods

The properties of the paper samples were evaluated in accordance with the following standards at Research Institute of Pulp and Paper Industry (RIPPI), Vietnam: basis weight (ISO 536), tensile strength (ISO 1924-2), moisture content (ISO 287), tear strength (ISO 1974), bursting strength (ISO 2758), folding endurance (ISO 5626), and thickness (ISO 534) after conditioning at 23 ± 1 °C and $50 \pm 2\%$ relative humidity (ISO 187).

Thermogravimetric analysis

The thermal degradation of the paper samples was investigated using thermogravimetric analysis (TGA). The samples were tested at a heating rate of 10 °C/min from 30 to 700 °C under a nitrogen atmosphere.

Scanning electron microscopy

The surface morphology of the paper samples was examined using a scanning electron microscope (SEM, JEOL JSM-7600F, Japan) at Hanoi University of Science and Technology.

Fourier transform infrared spectroscopy

FTIR analysis was performed using an FTIR spectrometer (Fisher, USA) at Hanoi University of Science and Technology, to identify the chemical functional groups of the paper samples. The spectra were recorded in the range of 4000–400 cm⁻¹ using the attenuated total reflectance (ATR) mode, with a resolution of 4 cm⁻¹ and 32 scans per sample.

RESULTS AND DISCUSSION

Heat-resistant paper for printed circuit board (PCB) applications requires specific mechanical and physical properties, including high basis weight, low thickness, and uniform fiber formation and distribution to ensure superior mechanical strength. To meet these requirements, the pulp must exhibit high purity, an appropriate degree of refining, well-developed fibrillation, and minimal fiber cutting.

Therefore, it is essential to investigate the processing conditions of the pulp, including refining degree, dosage of chemical additives, and surface treatment conditions of the paper.

Effects of acid purification on the properties of pulp

Paper substrates intended for printed circuit board applications require a high degree of cleanliness and minimal impurity content. Therefore, the pulp was subjected to a purification treatment prior to further processing to remove inorganic contaminants. Based on previous studies, the purification process was performed using an inorganic acid (H_2SO_4), with the reaction pH adjusted to 4.0–4.5 to promote the release and removal of inorganic impurities (ash content) from the fiber matrix. A series of experiments was conducted under controlled laboratory conditions at ambient temperature. The pulp consistency was maintained at 10%, and the treatment time was fixed at 60 min. The acid dosage was varied from 1% to 4% based on the oven-dry pulp mass. The physicochemical properties of the purified pulp were subsequently analyzed, and the results are summarized in Table 1.

The results in Table 1 show that increasing the H_2SO_4 dosage reduced the ash content from 0.63% to 0.36%, indicating effective removal of inorganic impurities. The extract pH remained close to neutral (7.11–7.30). The tensile strength decreased slightly with increasing acid dosage (from 6.55 to 6.16 kN/m at 4%), probably because of mild acid hydrolysis of cellulose, whereas the tear strength remained almost unchanged. An acid dosage of 2% (ash content 0.40%, tensile strength 6.22 kN/m) provides a balance between pulp purity and strength and was therefore selected for subsequent experiments.

Effects of refining degree on paper properties

Refining degree is one of the key technological parameters that directly affects fiber structure and the properties of the final paper. For kraft pulp, the refining process modifies fiber morphology through mechanisms such as fibrillation, fiber shortening, and fiber wall softening. These changes significantly influence the structural density, porosity, and drainage behavior of the pulp, which are closely related to the mechanical properties of the resulting paper. Based on these requirements, a series of experiments were conducted with refining degrees ranging from 35 to 55 °SR, a basis weight of $160 \pm 5 \text{ g/m}^2$, and 0.5% strength additive. The analytical results are presented in Table 2.

Table 1
Analytical results of purified pulp samples

No	Parameter	Unit	H_2SO_4 dosage, %				
			0	1	2	3	4
1	Extract pH	-	7.11	7.21	7.3	7.15	7.23
2	Ash content	%	0.63	0.52	0.4	0.39	0.36
3	Tensile strength	kN/m	6.55	6.23	6.22	6.22	6.16
4	Tear strength	mN	790	786	775	770	790

Table 2
Properties of the paper at different refining degrees

No	Parameter	Unit	Degree, °SR				
			35±2	40±2	45±2	50±2	55±2
1	Basis weight	g/m^2	160.7	160.6	160.3	161.3	160.9
2	Tensile strength	kN/m	6.46	8.15	8.77	9.16	9.25
3	Tear strength	mN	1860	1877	2120	2310	2265
4	Thickness	mm	0.254	0.225	0.225	0.221	0.218

At a low refining degree, limited fibrillation resulted in insufficient inter-fiber bonding, leading to poor sheet formation and reduced internal bonding strength. Increasing the refining degree from 35 to 50 °SR enhanced fibrillation, thereby increasing the contact area between fibers and significantly improving mechanical strength and structural uniformity. Consequently, the tensile strength increased from 6.46 to 9.25 kN/m and the tear strength from 1860 to 2310 mN. However, further increasing the refining degree to 55 °SR led to excessive fiber shortening, resulting in only marginal improvement in tensile strength and a decline in tear strength. In addition, higher refining levels require longer processing

time and greater energy consumption. Therefore, an optimal refining degree of 40 ± 2 °SR was selected for subsequent experiments.

Effects of the strength additive on the fiber network (SEM analysis)

The SEM images (Fig. 2) reveal a clear difference in fiber morphology and network structure between paper samples with and without the strength additive. In the untreated sample (without additive), the fiber network appears loosely packed with visible voids and irregular fiber distribution. The fiber surfaces are relatively rough, and the contact between fibers is limited, indicating weak inter-fiber bonding.

In contrast, the sample containing the strength additive exhibits a more compact and uniform structure. The fibers are more closely packed, with reduced porosity and increased contact areas. A thin film-like layer can be observed on the fiber surfaces, which is attributed to the deposition of the additive. This layer enhances fiber–fiber adhesion and promotes the formation of a more continuous and integrated network.

These structural changes at the micro-scale are consistent with the observed improvements in mechanical properties, particularly tensile strength, confirming the role of the strength additive in reinforcing the fiber network.

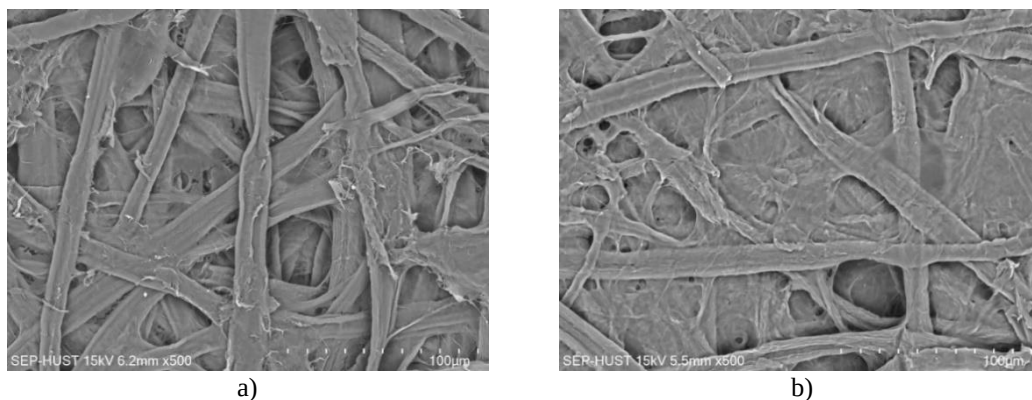


Figure 2: SEM images of paper sheets (a) without and (b) with strength additives

Effects of surface treatment on the thermal stability of paper

Native cellulose exhibits limited thermal stability and is inherently flammable, with degradation typically starting at temperatures above 200 °C. These limitations hinder its application in electronic systems that require thermal resistance and fire safety. To address these challenges, bio-based heat-resistant coatings have been developed to enhance the thermal performance of paper substrates. These coatings not only improve flame retardancy, but also provide barrier properties and mechanical reinforcement. Surface treatment of the paper was carried out using a coating formulation consisting of a base polymer, crosslinking agent, catalyst, and water, in a ratio of 85:11.7:3.3:220, corresponding to a solid content of 15%. The basis weight of the paper was 160 g/m². The coating process was performed using a laboratory-scale coating device.

The surface treatment system, consisting of polymer emulsion, crosslinking agent, and catalyst, forms a functional coating layer on the paper surface. During application, the polymer penetrates into the fiber network and fills inter-fiber voids. Upon thermal curing, a three-dimensional crosslinked polymer network is formed, generating a continuous film strongly adhering to the cellulose fibers. This structure enhances mechanical strength, reduces porosity, and improves moisture and thermal resistance. Additionally, the low surface energy of the coating imparts anti-adhesion properties, while the crosslinked network ensures stability under elevated temperature and humid conditions.

The thermal stability of the surface-treated paper was evaluated using TGA/DTG analysis (Fig. 3). The results show that the onset decomposition temperature (T_{onset}) reached 301.8 °C, indicating good resistance to initial thermal degradation. The main decomposition stage occurred between 301.8 and 385.4 °C, with a maximum degradation rate (T_{max}) at 316.5 °C, as observed from the DTG peak.

The significant mass loss of 84.66% in this region corresponds to the degradation of the cellulose backbone, while the initial minor weight loss (7.38%) is attributed to moisture evaporation and the

release of low-molecular-weight compounds. Notably, a residual mass of approximately 2.21% remained at high temperatures, suggesting the formation of a stable char structure.

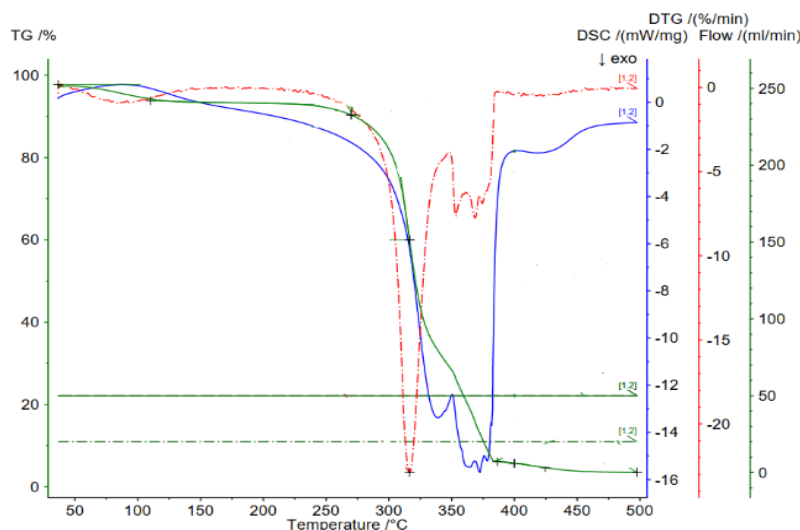


Figure 3: Thermogravimetric analysis of thermally resistant paper

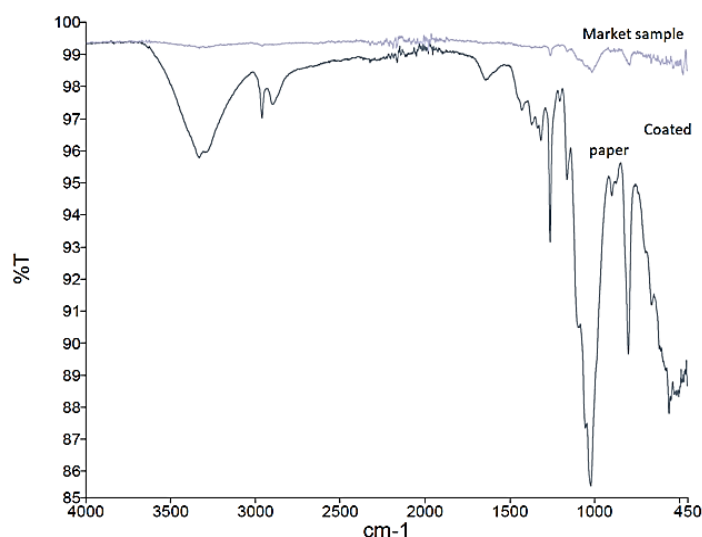


Figure 4: FTIR spectra of the market sample and coated paper

The relatively high T_{onset} and T_{max} values, along with the presence of residual char, indicate that the surface treatment contributes to enhanced thermal stability. This improvement can be attributed to the formation of a crosslinked polymer network on the fiber surface, which acts as a protective barrier, limiting heat transfer and slowing down the release of volatile degradation products.

Overall, the TGA/DTG results confirm that surface modification effectively delays thermal decomposition and promotes char formation, thereby improving the heat resistance of paper.

In Figure 4, the FTIR spectra of the market sample and coated paper reveal distinct differences in chemical structure. The broad absorption band observed around 3200–3500 cm^{-1} in both samples corresponds to the stretching vibration of hydroxyl ($-\text{OH}$) groups, characteristic of cellulose. However, this peak appears less intense in the coated sample, suggesting reduced hydrogen bonding due to surface modification. The absorption peaks near 2900 cm^{-1} are attributed to C–H stretching vibrations of aliphatic groups. In the coated paper, additional prominent peaks are observed in the region of 1000–1100 cm^{-1} , which are assigned to Si–O–Si stretching vibrations, confirming the presence of a silicone-based coating layer. Furthermore, the coated sample exhibits stronger and more defined peaks in the 800–900 cm^{-1} region, associated with Si–C and Si–O functional groups, indicating successful deposition of the polymer on the paper surface. In contrast, the market sample shows weaker or absent signals in these regions, reflecting the absence or lower content of silicone structures. Overall, the FTIR results

confirm that surface treatment introduces functional groups onto the paper surface, leading to structural modification and contributing to improved thermal stability and anti-adhesion properties.

Mechanical properties of coated paper

The mechanical properties of the uncoated and coated papers are summarized in Table 3. The coated paper reached a tensile strength of 8.31 kN/m, a tear strength of 1955 mN, a bursting strength of 792 kPa, a folding endurance of 800 double folds. Compared with the uncoated paper, the coating changed these values by 2-4%, because the crosslinked polymer film fills the inter-fiber voids and bonds the surface fibers. After exposure to 250 °C for 30 min, the coated paper retained 97.6% of its tensile strength and 95.25% of its folding endurance, which indicates that it can withstand the hot-pressing conditions of PCB lamination. Figure 5 presents macroscopic photographs of the heat-resistant paper developed in this study: (a) the paper sheets before and after coating, and (b) the coated sheet before and after heat treatment at 250 °C for 30 min.

Table 3
Mechanical properties of uncoated and coated papers

Property	Standard	Uncoated paper	Coated paper	Coated paper after heat exposure
Tensile strength, kN/m	ISO 1924-2	8.15	8.31	8.11
Tear strength, mN	ISO 1974	1877	1955	1891
Bursting strength, kPa	ISO 2758	786	792	765
Folding endurance, double folds	ISO 5626	795	800	765
Moisture content, %	ISO 287	6.1	6.5	5.7



Figure 5: Photographs of (a) uncoated and coated heat-resistant paper, (b) coated paper before and after heat exposure

CONCLUSION

Based on the obtained results, a technological process for the production of heat-resistant paper for printed circuit board applications was successfully established. The process involves acid pretreatment with 2% H₂SO₄, refining to 40 °SR, incorporation of 0.5% dry-strength additive, and subsequent surface treatment with a polymer coating. The resulting paper had a tensile strength of 8.31 kN/m, a tear strength

of 1955 mN, and thermal stability up to about 300 °C. At the end of its life, the paper can be recycled into other paper products, such as industrial packaging paper, because the silicone coating accounts for only about 2.5% of its mass. The product has been pilot-produced at the Testing Center of the Institute of Pulp and Paper Industry and is currently in the stage of market introduction and product promotion. This study provides a practical and scalable approach for enhancing the thermal and mechanical performance of paper through a synergistic combination of chemical treatment and surface modification, offering a promising candidate material for high-performance and sustainable paper-based electronic substrates. Future work will focus on fabricating circuit boards with this paper and testing them under real PCB manufacturing conditions.

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