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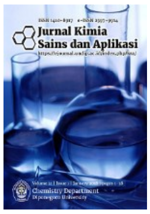

Evaluation of Sealing Strength in Multilayer LLDPE Packaging for Impact-Resistant Rice Pouches

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Abstract

In this study, we evaluate the mechanical integrity, thermal stability, and impact resilience of three different laminated polymer materials—PA/LLDPE, MDO-PE/LLDPE, and BOPE/LLDPE—to assess their suitability for rice packaging applications. Our experiments include tests to determine the temperature at which sealing begins, evaluations of thermal durability, assessments of seal integrity, and impact resistance tests. PA/LLDPE exhibited excellent sealing ability, displaying the highest seal strength of 6,411 gr/15 mm at 150°C, and maintained structural integrity up to 200°C, making it particularly suitable for high-temperature applications. MDO-PE/LLDPE also performed well, especially at lower temperatures, with a seal strength of 5,327 gr/15 mm at 130°C. The addition of a plastomer significantly improved its low-temperature effectiveness. Conversely, BOPE/LLDPE reached its peak seal strength at 3,825 gr/15 mm at 140°C but showed lower stability at higher temperatures. During drop tests, PA/LLDPE and MDO-PE/LLDPE demonstrated good impact resistance, absorbing up to 245.250 Joules, while BOPE/LLDPE absorbed only up to 196.240 Joules, indicating its limited ability to withstand impact. These results suggest that PA/LLDPE and MDO-PE/LLDPE are more effective in preventing environmental ingress, even under stress at temperatures up to 200°C. This research underscores the critical role of polymer composition and structure in enhancing the mechanical performance and durability of packaging materials. The practical applications of these findings are substantial, offering packaging developers insights into selecting appropriate materials that meet industry standards for safety and environmental responsibility, particularly in the food sector. These insights could lead to the development of more sustainable, high-performance packaging solutions, aligning with global trends toward sustainability and safety in food packaging.

1. Introduction

The sealing strength of pouch packaging is a critical parameter that directly influences its products' safety, freshness, and shelf life. This attribute describes the seal's ability to withstand stress and remain intact under various conditions such as handling, transportation, and storage [1]. An effective seal prevents contaminants like oxygen, moisture, and microorganisms from entering, which could compromise product quality and safety. It also maintains a protective atmosphere inside the package—often used to extend product freshness—without leakage. Advanced forms of polyethylene, including PA/LLDPE (Polyamide/Linear Low-Density Polyethylene), MDO-PE/LLDPE (Machine Direction Oriented Polyethylene/Linear Low-Density Polyethylene), and BOPE/LLDPE (Biaxially Oriented Polyethylene/Linear Low-Density Polyethylene), play a pivotal role in the packaging industry. These materials are selected for their superior mechanical properties, such as flexibility, strength, and resistance to puncture and tear [2]. Additionally, they provide excellent clarity and gloss, enhancing the aesthetic appeal crucial for consumer products.

PA/LLDPE layers are especially valued for their barrier properties. Polyamide acts as an effective barrier against oxygen and aromas, preserving the freshness and flavor of food products. When laminated with LLDPE, the material gains structural integrity and enhanced sealability, essential for maintaining package hermeticity [3, 4]. This combination is commonly used in food packaging to prolong shelf life and protect sensitive products from environmental factors [5, 6]. MDO-PE/LLDPE represents a significant advancement in polyethylene technology. The orientation process aligns the polymer chains in the machine direction, increasing stiffness and strength while preserving flexibility and sealing characteristics. This structure suits high-speed packaging applications, providing improved mechanical properties that withstand the demands of modern packaging, distribution, and retail systems [7].

BOPE/LLDPE is an innovative approach where polyethylene is biaxially oriented, enhancing both mechanical and barrier properties. This material reduces packaging film thickness without sacrificing performance, offering environmental benefits through material reduction and enhanced logistics efficiency [3, 4, 8]. Its excellent optical and mechanical properties are ideal for high-quality printing and robust packaging solutions, which are crucial for both consumer appeal and functional performance. The demand for such advanced polyethylene materials is driven by the need for sustainable packaging solutions that minimize environmental impact while enhancing performance characteristics vital for modern supply chains. These materials enable the production of lighter, stronger, and more recyclable pouches, aligning with global sustainability goals [9, 10]. In conclusion, the sealing strength of pouch packaging is fundamental to ensuring the integrity and quality of packaged goods [11].

Materials such as PA/LLDPE, MDO-PE/LLDPE, and BOPE/LLDPE are at the forefront of this endeavor, offering tailored properties to meet the specific demands of diverse packaging applications [12]. Their ongoing development signifies continuous innovation in material science aimed at optimizing the safety, efficiency, and sustainability of packaging solutions in a complex global market. The continued advancement of these materials is essential for meeting the evolving needs of both consumers and industries, ensuring that products reach consumers in the best possible condition [13].

2. Experimental

2.1. Materials, Laboratory Apparatus, and Instrumentations

Laminate films were produced using dry lamination techniques that combined with an 80-micron thick layer of LLDPE, specifically LLDPE C6, with layers of polyamide (PA), machine-direction oriented polyethylene (MDO-PE), and biaxially oriented polyethylene (BOPE). This composition included LLDPE enriched with LLDPE C6 and plastomer resin, a formulation tailored to enhance the visual quality of seals and prevent wrinkling in MDO-PE and BOPE films during heat sealing. The lamination process was carried out using the DL-03 machine, with the lamination chamber maintaining a precise temperature range of 70-80°C to ensure optimal adhesive conditions.

The standardized coating weight was set at 3.0 g/m², and the temperature of the laminating rolls was consistently held between 45°C and 50°C to promote uniform lamination. PA, MDO-PE, and BOPE films intended for lamination were subjected to a surface treatment to achieve a wettability of 38 dyne/cm². This was accomplished using a dyne marker with AFS® equipment, which marked the designated substrate with a specific contact time characterized by a thickness of 25 µm and a density of 1.14 g/cm³, 0.930 g/cm³, and 0.925 g/cm³. Samples were prepared for this study to test the laminated structure as described in Table 1.

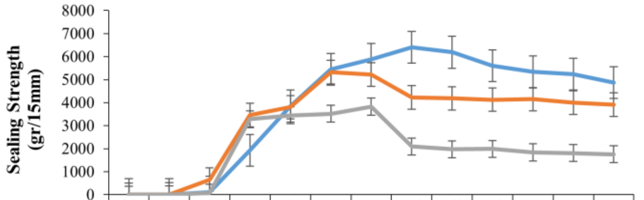
Seal strength tests were carried out on three samples of laminated structures using a sealing process [12, 13]. The parameters for these tests followed ASTM F88 standards, with samples measuring 15 × 100 mm in width and height. The testing was conducted in an unsupported manner, with a clamping distance of 10 mm and a pulling speed of 300 mm per minute. Seal strength was calculated by dividing the maximum force applied by the seal width (15 mm), and it exhibited variations with temperature fluctuations.

For each sealing temperature condition, a set of ten samples was tested using a Brugger© HSG-CC sealer machine with a sealing force ranging from 40 N to 1000 N. The dwell time was limited to 0.1 – 300 seconds, and the table devices had dimensions of 65 × 35 × 44 cm with sealing tolerances of 1.5°C + 10%, manufactured by Brugger-Feinmechanic.

A Universal Tensile Machine (UTM) Vantage-NX was utilized, measuring 61 cm × 36 cm × 48 cm, with a clamping force of up to 3000 N. It was connected to a personal computer for data analysis. The JDC Cutter sampler from Thwing Albert was used to cut samples into 15 mm widths. Hermeticity testing involved a 100 g weight, a dwell time of 0.5 seconds, and a sealing pressure of 150 N, with temperatures ranging from 100 to 180°C.

Table 1. Description of laminated films for the study

No.	Laminated structure	Overall thickness (micron)	Grammature (kg/m²)
1	PA 25 / LLDPE 80	105	0.0890
2	MDO-PE 25 / LLDPE 80	105	0.0944
3	BOPE 25 / LLDPE 80	105	0.0943



	80	90	100	110	120	130	140	150	160	170	180	190	200
PA/LLDPE	0	12	115	1932	3851	5447	5887	6411	6187	5609	5332	5243	4866
MDO-PE/LLDPE	0	14	653	3455	3801	5327	5220	4223	4184	4128	4148	4001	3912
BOPE/LLDPE	0	10	84	3279	3449	3514	3825	2092	1974	1990	1838	1811	1759

Figure 1. Sealing initiation temperature of PA/LLDPE, MDO-PE/LLDPE, and BOPE/LLDPE

2.2. Preparation of Sample Sealing Initiation Temperature (SIT) and Hermeticity Seal

Samples were tested individually at temperatures from 80°C to 200°C to determine the sealing initiation temperature. Each sample was cut to a dimension of 15 mm × 100 mm and sealed using a sealer machine 150 N pressure for 0.5 seconds. To ensure integrity, measuring any displacement in the sealing area using a ruler to assess the seal's effectiveness is important. The hermeticity of the seals was evaluated by checking the tightness of the sealing position and observing the phenomena during tensile testing with Thwing Albert©.

2.3. Preparation of Sample Heat Resistances

To study the film's heat resistance and behavior under specific conditions, the sample preparation involved applying it to substrates between 80°C and 200°C. Each sample, measuring 30 mm × 50 mm, was sealed using a machine that applied pressure of 150 N for 0.5 seconds, with pressure and dwell time maintained as constant variables.

2.4. Fabrication Pouch for Free-Fall Test Simulation

Pouch packaging for PA/LLDPE, MDO-PE/LLDPE, and BOPE/LLDPE was produced using the TTN-01 machine, developed by a Japanese manufacturer, with pouch dimensions set at 300 mm × 450 mm. The machine operated with a sealing temperature range of 140°C to 180°C, a pressure range of 3 to 5 bar, and a production rate of 50 pieces per minute. It could produce pouches within a 75 mm to 600 mm size range and a seal width of 25 mm to 50 mm. The durability of the materials was tested through a free-fall test, dropping pouches from heights of 1 to 5 meters, with weights of 1 to 5 kg, to assess their impact energy that could potentially cause damage to the pouches. The rice used for this drop testing was obtained from a local Indonesian brand.

3. Results and Discussion

3.1. Sealing Initiation Temperature (SIT) and Hermeticity Seal for MDO-PE/LLDPE, BOPE/LLDPE, and PA/LLDPE

Seal strength plays a crucial role in the packaging industry [14, 15, 16], especially when evaluating the effectiveness of laminated layers like PA/LLDPE, MDO-PE/LLDPE, and BOPE/LLDPE. It is essential for determining the layers' ability to maintain a secure seal and withstand pressures or deformations [17]. The seal strength in these combinations is significantly influenced by the mechanical properties of each polymer and their interactions during the sealing process.

As illustrated in Figure 1, the PA/LLDPE material demonstrates an initial seal strength of 0 gr/15 mm at 80°C, indicating no adhesion or seal formation at this temperature. However, when the temperature increases to 90°C, the seal strength significantly increases to 12 gr/15 mm. A significant increase occurs between 100 and 110°C, where the seal strength increases from 115 gr/15 mm to 1,932 gr/15 mm. This upward trend continues, peaking at 150°C with a seal strength of 6,411 gr/15 mm, indicating a very strong seal at this temperature.

Beyond this, seal strength is decreased, dropping to 4,866 gr/15 mm at 200°C. This reduction may be attributed to thermal degradation at higher temperatures. PA/LLDPE: polyamide is prone to thermal degradation due to the breakdown of its amide groups (-CONH-). Elevated temperatures can induce reactions such as dehydration (water loss), leading to chain scission, which diminishes both molecular weight and mechanical strength [8]. MDO-PE/LLDPE: MDO-PE, a variant of high-density polyethylene, typically exhibits a high degree of crystallinity and a more ordered structure, which confers a certain level of thermal stability. Nonetheless, at elevated temperatures, thermal oxidation can occur, resulting in either chain scission or cross-linking, thereby compromising the material's structural integrity [18, 19]. BOPE/LLDPE: BOPE, similar to MDO-PE but oriented in both machine and transverse directions, exhibits enhanced crystallinity and molecular orientation. While this structure imparts significant thermal stability, it remains susceptible to thermal oxidation and degradation at high temperatures [1].

The MDO-PE/LLDPE material, starting at 14 gr/15 mm at 80°C, already exhibits better adhesion at lower temperatures than PA/LLDPE. There is a sharp increase to 653 gr/15 mm at 90°C, continuing to rise to 5,327 gr/15 mm at 140°C. Despite minor fluctuations, the seal strength of this material remains relatively stable between 140 and 180°C, demonstrating a broad operational sealing range. Like PA/LLDPE, a decline in seal strength is noted after 180°C, decreasing to 3,912 gr/15 mm at 200°C, which can also be interpreted as an effect of thermal degradation. In the case of BOPE/LLDPE, the initial seal strength is 10 gr/15 mm at 80°C, steadily increasing to 3,449 gr/15 mm at 120°C. This increment continues, albeit more smoothly than the other two materials, reaching 3,825 gr/15 mm at 140°C.

Table 2. Heat resistance comparison between PA/LLDPE, MDO-PE/LLDPE, and BOPE/LLDPE

Applied temperature (°C)	PA/LLDPE	MDO-PE/LLDPE	BOPE/LLDPE
80	O	O	O
90	O	O	O
100	O	O	O
110	O	O	O
120	O	O	O
130	O	O	O
140	O	O	O
150	O	Δ	Δ
160	O	Δ	Δ
170	O	X	X
180	O	X	X
190	O	X	X
200	O	X	X

(O) = Good, (Δ) = Wrinkle, (X) = Broken

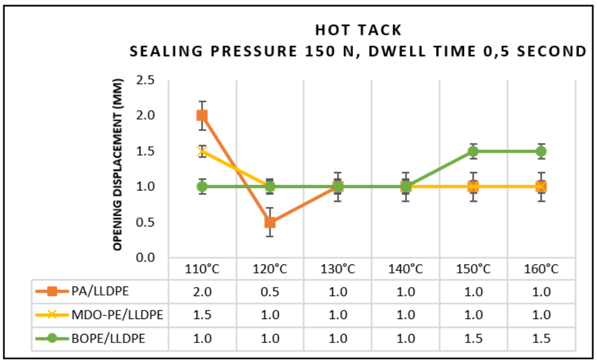


Figure 2. Hot tack performances (hermeticity seal) PA/LLDPE vs. MDO-PE/LLDPE vs. BOPE/LLDPE

Interestingly, the seal strength of BOPE/LLDPE shows a consistent decline after reaching 130°C, falling to 1,759 gr/15 mm at 200°C. This decrease may indicate that BOPE/LLDPE is sensitive to higher temperatures and lacks the same thermal stability as the other materials. This data shows that each polymer material exhibits unique characteristics related to its seal strength, which varies according to the sealing temperature. PA/LLDPE demonstrates strong sealing performance at mid to high temperatures but is susceptible to degradation at higher temperatures. MDO-PE/LLDPE exhibits better initial adhesion and seal strength stability across a broader temperature range, making it suitable for sealing applications that require lower temperatures.

Polyamide exhibits a higher melting range than MDO-PE and BOPE (Table 3), which melt between 119–138°C. This elevated melting range of polyamide is attributed to hydrogen bonding, with each repeating unit forming four hydrogen bonds. Specifically, the two amide hydrogen atoms and the two carbonyl oxygen atoms within each repeating unit of polyamide engage in hydrogen bonding. The absence of secondary hydrogen bonding in MDO-PE or BOPE contributes to its comparatively lower melting range. These secondary hydrogen bonds significantly increase the energy required to disrupt the intermolecular forces, preventing the polymer chains from sliding past each other.

Figure 3. Illustration of heat resistances testing PA/LLDPE, MDO-PE/LLDPE, and BOPE/LLDPE

For MDO-PE/LLDPE and BOPE/LLDPE, the addition of polyolefin plastomers (POP) material enhances the sealing capabilities at lower temperatures and improves seal strength. This effect is due to the metallocene-catalyzed PE grades, which, with high comonomer content, are classified as VLDPE (very low-density polyethylene) due to their low density (0.89–0.91 g/cm^3) and many side branches. These grades are also referred to as POP because they incorporate rubber-like properties alongside their thermoplastic characteristics [20, 21]. The data highlights the significant impact of temperature on seal integrity, especially for PA/LLDPE. The significant reduction in opening displacement between 110°C and 120°C for PA/LLDPE signifies a crucial threshold where the material properties stabilize, possibly influenced by the thermal behavior of the polyamide and LLDPE combination [22]. Conversely, the more uniform hot tack performance of MDO-PE/LLDPE and BOPE/LLDPE across the tested range suggests that these materials are less prone to performance fluctuations due to temperature variations during the sealing process. This characteristic makes them suitable for processes where temperature control might vary slightly, providing a larger window for achieving optimal seal conditions.

3.2. Heat Resistances for PA/LLDPE, MDO-PE/LLDPE, and BOPE/LLDPE

Heat resistance is a measure of the thermal endurance of plastic materials, indicating the specimen's ability to maintain its shape under specific temperature and load conditions or to deform within set limits at a defined test temperature, typically ranging from 80 to 200°C. The comparison of heat resistance characteristics among the samples is presented in Table 2.

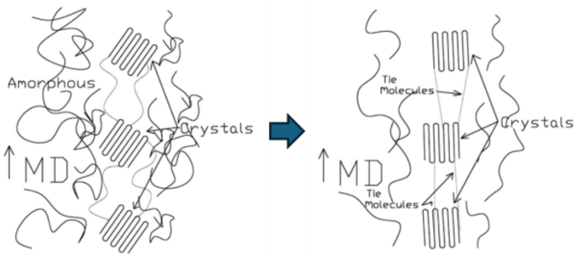


Figure 4. Mechanism of polymer molecular alignment [4]

The dataset reveals a discernible trend: the structural integrity of each film decreases as temperature increases, although the thermal thresholds vary among the different material composites. All three films exhibit good integrity at temperatures ranging from 80°C to 150°C (denoted as 'O'). However, each film begins to deform and fail to different extents above this range. As shown in Figure 3, during the heat-sealing process, two sealer jaws—upper and lower—are used to apply heat and pressure to the films, simulating conditions that assess the thermal stability and sealability of packaging materials. The outer layer of the films can be composed of different materials such as PA, MDO-PE, or BOPE, while the inner layer consists of LLDPE as the sealing layer.

The PA/LLDPE film demonstrates superior thermal resistance, maintaining its structural integrity up to 200°C. This resilience is likely due to the inherent properties of polyamides—known for excellent thermal stability—and their use in high-temperature applications. Additionally, integrating LLDPE, which provides toughness and flexibility, likely enhances the film's resistance to heat-induced stress, thereby preventing failure up to the highest tested temperature. Conversely, the MDO-PE/LLDPE film exhibits initial wrinkling (indicated by '∆') at 150°C, progressing to breakage (indicated by 'X') at 170°C.

This behavior may stem from the film's machine direction orientation (MDO), which aligns polymer chains longitudinally, increasing strength and stiffness along the machine direction but potentially reducing tensile strength and thermal resistance transversely, leading to the noted failures at elevated temperatures. In this context, molecular alignment can enhance the material's strength through the orderly arrangement of molecules, as illustrated in Figure 4.

Table 3. DSC analysis comparison between PA, MDO-PE, and BOPE

Sample		Thermal properties (Tm°C)		
		Polyamide	MDO-PE	BOPE
First heating peak data	Onset	216.06	121.33	126.69
	Peak	223.50	131.32	130.78
	Endset	228.97	137.03	134.30
Second heating peak data	Onset	205.90	120.46	119.35
	Peak	219.90	132.85	124.78
	Endset	225.25	138.05	130.55

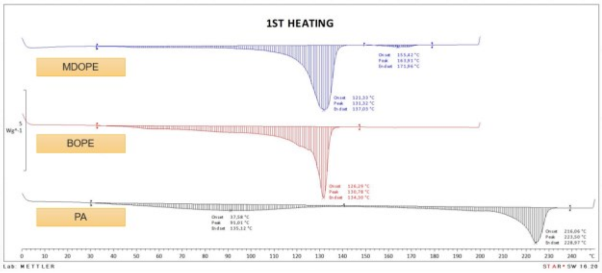


Figure 5. DSC analysis of outer layer PA, MDO-PE, and BOPE: 1st heating cycle

Heating the film increases polymer molecule mobility, allowing for higher draw ratios and improved film properties, with orientation temperature playing a key role. Various theories explain molecular changes during stretching; one, originally for HDPE, also extends to other polymers. Before MDO orientation, the film has ordered crystalline regions connected by molecules traversing surrounding amorphous areas. These regions contain randomly arranged molecules, while the crystalline plates have molecules tilted at 34.4° relative to the film surface [1]. During stretching in the machine direction (MD), the randomly oriented amorphous molecules begin to align with the direction of orientation. This alignment process continues until the molecules linking the crystals are fully extended [4].

Similarly, the BOPE/LLDPE film begins to wrinkle at 150°C and breaks at 170°C. Its biaxial orientation, stretching the film in longitudinal and transverse directions, generally enhances barrier properties and mechanical strength. However, this orientation may also induce internal stresses, diminishing thermal resistance, as evidenced by the similar performance degradation observed in the MDO-PE/LLDPE film. This comparative analysis underscores the influence of specific manufacturing processes and polymer molecular orientation on the thermal resistance of films.

The findings confirm that PA/LLDPE films outperform MDO-PE/LLDPE and BOPE/LLDPE in maintaining integrity at higher temperatures. The thermal data provided for polyamide, MDO-PE, and BOPE reveal significant variations in their thermal properties in Table 3 and Figures 5 and 6. Through differential scanning calorimetry (DSC) analysis, the melting temperatures of each material can be compared, serving as crucial indicators of thermal stability and potential applications in product designs that require high sealing temperatures and extreme temperature conditions.

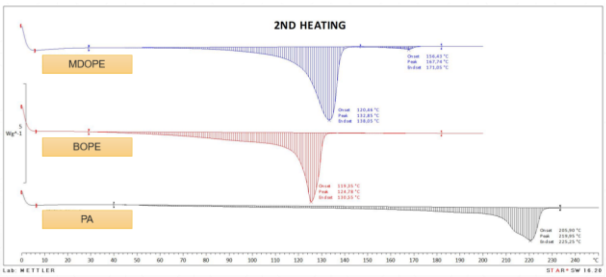


Figure 6. DSC analysis of outer layer PA, MDO-PE, and BOPE: 2nd heating cycle

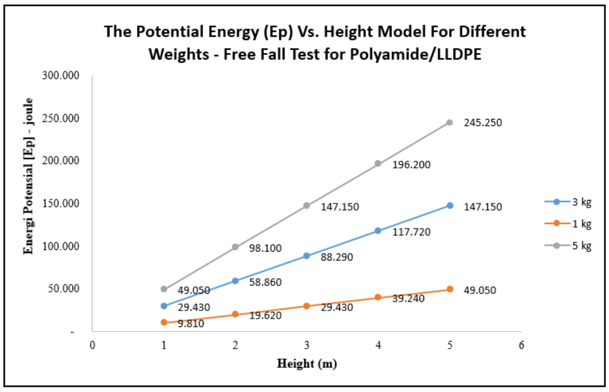


Figure 7. Potential energy (Ep) vs. height model for different weights – PA/LLDPE

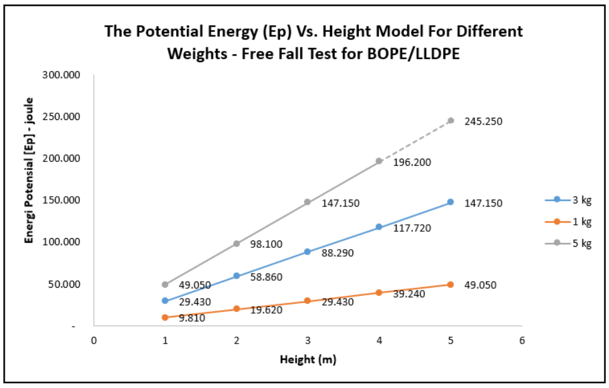


Figure 8. Potential energy (Ep) vs. height model for different weights – BOPE/LLDPE

Polyamide, known for its exceptional physical and thermal properties, showed a melting onset of 216.06°C when heated initially, peaking at 223.50°C and ending at 228.97°C (Figure 5). Upon the second heating cycle, the onset commenced at 205.90°C, peaked at 219.90°C, and concluded at 225.25°C. In contrast, MDO-PE, recognized for its resilience and flexibility, demonstrated a notably lower melting onset temperature of 121.33°C during the initial heating phase, peaking at 131.32°C and concluding at 137.03°C. Subsequently, in the second heating cycle, the onset temperature was 120.46°C, peaked at 132.85°C, and concluded at 138.05°C (Figure 6).

3.3. Fabrication Pouch for Free-Fall Test Simulation

The manufacturing process of pouches in the flexible packaging industry is a complex and meticulous operation essential for producing high-quality and durable packaging solutions. Once the appropriate materials are selected, the pouch-making machine is precisely configured to specific dimensions. The edges of the pouch are sealed by passing the material through heating elements. Sealing is the process of fusing two materials to merge to form a pouch.

The potential energy (Ep) vs. height model for different weights in a free-fall test is utilized to analyze the energy characteristics of objects exposed to gravitational forces, which is crucial when evaluating the impact resistance of packaging in drop tests. This model is based on the principle that potential energy increases directly to height and mass. Mathematically, it is represented as Ep = mgh, where m stands for the mass, g represents the acceleration due to gravity (approximately 9.81 m/s^2), and h denotes the height from which the object is released. In the context of a free fall test, various weights can be examined to ascertain how mass influences the performance and durability of packaging upon impact.

A higher mass generally means greater potential energy at a given height, which can lead to more significant forces upon impact. This modeling helps in designing packaging that can withstand the potential forces during transportation, ensuring that the integrity of the contents is maintained. Understanding these dynamics allows manufacturers to optimize packaging materials and designs to suit various weight categories and drop scenarios. Figures 7, 8, and 9 demonstrate that packaging utilizing PA/LLDPE and MDO-PE/LLDPE can absorb energy from 196.240 to 245.250 Joules. In contrast, BOPE/LLDPE packaging can only absorb a maximum of 196.240 Joules. Furthermore, these materials exhibit different types of defects, indicating varied energy absorption capacities depending on the structural characteristics of the materials tested.

3.4. Failure Analysis of Sealing Strength in BOPE/LLDPE Laminates

As demonstrated in the free-fall tests, PA/LLDPE effectively absorbs energy. However, BOPE/LLDPE shows different results; after a 5-meter drop with a 5 kg rice volume, BOPE/LLDPE experiences delamination between layers, compromising seal integrity. Figure 10(a) reveals that BOPE undergoes cohesive failure, leading to leakage in the tested material. This is further illustrated in Figure 10(b), where the presumed cause is the inability of the BOPE/LLDPE structure to withstand impacts from certain heights, resulting in the spontaneous separation of the BOPE/LLDPE layers upon impact.

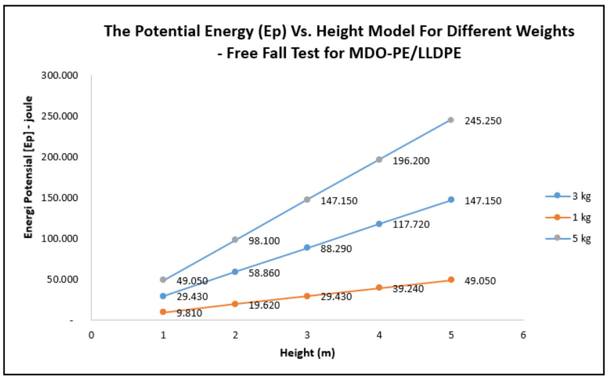


Figure 9. Potential energy (Ep) vs. height model for different weights – MDO-PE/LLDPE

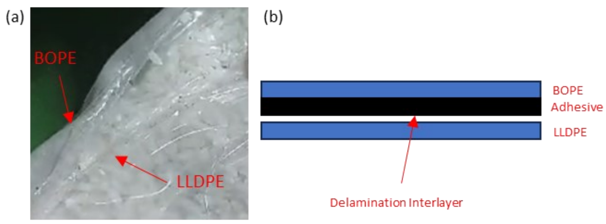


Figure 10. (a) Visual evidence of interlayer delamination in BOPE/LLDPE, (b) Schematic illustration of interlayer delamination in the structure of BOPE/LLDPE

4. Conclusion

This study provided a detailed analysis of the sealing strength, thermal stability, and impact resistance of three laminate polymers: PA/LLDPE, MDO-PE/LLDPE, and BOPE/LLDPE, tailored for use in flexible packaging. PA/LLDPE laminates demonstrated superior thermal resistance, maintaining structural integrity with peak sealing strengths reaching 6,411 gr/15 mm at 150°C and effectively withstanding temperatures up to 200°C. This makes PA/LLDPE highly suitable for high-temperature applications with critical durability. MDO-PE/LLDPE showed robust sealing capabilities at lower temperatures, achieving a maximum seal strength of 5,220 gr/15 mm at 140°C. The inclusion of the plastomer notably enhanced its sealing performance below 100°C, expanding its application range to conditions requiring moderate thermal resistance but excellent mechanical integrity. Conversely, BOPE/LLDPE reached its sealing strength apex at 3,825 gr/15 mm at 140°C but exhibited reduced stability at higher temperatures, limiting its use to applications with less thermal stress. Hermeticity tests further confirmed that all materials provided effective barriers against external contaminants yet highlighted the superior barrier properties of PA/LLDPE and MDO-PE/LLDPE at elevated temperatures. Impact tests revealed that PA/LLDPE and MDO-PE/LLDPE could absorb up to 245.250 Joules, demonstrating significant impact resistance, while BOPE/LLDPE managed only up to 196.240 Joules, indicating a lesser ability to absorb shock under similar conditions.

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