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Development and Characterization of Polyisobutylene Based Rodent Adhesive Gel: Effect of Formulation on Adhesion, Shear Resistance and Viscosity

Hanan Sadiq[1], Abdul Ghaffar[2]
*(Chemistry, University of Engineering and Technology, Lahore
Email: hanansadiq463@gmail.com)
** (Chemistry, University of Engineering and Technology, Lahore
Email: geoghafar78@gmail.com)

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Abstract:

Rodent infestation is a continuous and serious problem in food protection, sanitation, agriculture and infrastructure and physical pest-management is needed. Polymeric adhesive systems offer an alternative way to immobilized rodents without potentially toxic active ingredients. Three polyisobutylene (PIB)-based adhesive gel formulations were prepared, the PIB and terpene resin concentrations were varied while the paraffin wax, talcum powder, butylated hydroxytoluene (BHT), and vegetable oil were held at the specified concentrations. The formulations were prepared by thermal processing (170–200 °C, 12 h), after which the vegetable oil was slowly added and cooled. The adhesive performance was tested by peel-type, shear failure and viscosity testing. The formulation without the terpene resin (F1) showed the lowest adhesive property and shear failure time of 30 min, whereas the formulations with terpene resin (F2 and F3) showed higher adhesive properties, measured as shear failure time of 120 and 150 min, respectively. The measured viscosities were 1.0 Pa·s, 1.5 Pa·s and 1.4 Pa·s for F1, F2 and F3 respectively. Thus, F3 presented the highest shear-failure time, but had a lower viscosity than F2. The results showed that viscosity was not the only factor that controlled adhesive performance, and that the composition of the formulation had a significant effect on the adhesion, flow behavior and resistance to sustained loading. The three formulations tested had the best combination of measured properties when formulated with F3. Based on the findings, further optimization of these adhesive systems is possible, and more extensive evaluation of PIB adhesive systems can be made in terms of rheology, durability and application.

Keywords — **polyisobutylene, rodent adhesive, adhesive gel, terpene resin, tackifier, shear resistance, viscosity, polymeric adhesive.**

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I. I. INTRODUCTION

Rodent infestation continues to be a problem in domestic, farm, industrial and storage facilities where they contaminate food and materials, damage structures and may transmit disease. Traditional control methods are exclusion, trapping and chemical control. While chemical rodenticides may be effective, there has been growing interest in physical control methods using adhesive immobilisation due to concerns from accidental exposure, environmental contamination and effects on non-target organisms.

Adhesive systems based on polymers combine several effects such as interfacial adhesion, tack, cohesive strength, resistance to deformation under load and viscosity, all of which contribute to the sustained adhesion. Polyisobutylene (PIB) is especially well suited as an adhesive matrix due to its hydrocarbon backbone, oleophobic properties and high viscosity. The adhesive property can be altered with the polymer composition, polymer properties and the rheological property. [1-4]

In addition to modifying the adhesive properties of polymers, tackifying resins can also be used to alter the surface tack and the relationship between the strength of the adhesion between the two phases and the cohesive strength. Of particular interest for this use are the terpene-based resins, which have been reported to affect the mechanical and adhesive properties of polymer systems. Formulation ingredients such as paraffin wax, talc, butylated hydroxytoluene (BHT), and vegetable oil may also help to impart consistency, physical characteristics, oxidative stability, and processing properties. [5-10]

Viscosity only partially controls adhesive performance. The desired adhesive system should have adequate surface adhesion and cohesive resistance to flow and failure under sustained loading. Thus, the evaluation of formulation performance must be taken in hand along with its shear failure resistance and viscosity. Recent research on PIB-based and terpene-based of pressure-sensitive adhesives also highlights the role of formulation composition on the rheological properties, adhesion and bond durability. [11-16]

Although there have been studies involving PIB-based adhesives and pressure-sensitive adhesives, few studies have been conducted on PIB-based adhesive gels that are manufactured specifically for physical rodent control. Specifically the correlation between the PIB and terpene-resin composition and the resultant adhesion, resistance to sustained loading and viscosity needs to be evaluated. [17-20] The present study thus compares three formulations of adhesive-gel based on PIB, which contain different proportions of PIB and terpene-resin but keep the other components of the formulations at certain levels. The evaluation of the adhesive performance, shear failure time, and viscosity was used to determine composition–performance relationships and to find the formulation that best matched the desired measured properties in the formulation space studied.

II. MATERIALS AND METHODS

A. Materials and Formulation Design

Polyisobutylene (M= 257,544.0212 g mol^{-1}) having Degree of polymerization 4599.000379, was employed as a major polymeric adhesive polymer and terpene resin (BT-911) was used as a tack-modifying agent. Supporting formulation ingredients were incorporated: paraffin wax, talcum powder, butylated hydroxytoluene (85%), and vegetable oil. Three formulations were prepared of varying proportions of PIB and terpene resin (F1, F2, F3) with the other formulation constituents at the same levels as indicated in Table I.

TABLE I. COMPOSITION OF PIB-BASED RODENT ADHESIVE-GEL FORMULATIONS.

Component	F1	F2	F3
PIB (g)	20	15	13
Terpene Resin (g)	0	5	7
Paraffin Wax (g)	5	5	5
Talcum Powder (g)	15	15	15
BHT (g)	0.5	0.5	0.5
Vegetable Oil (ml)	10	10	10

B. Adhesive Gel Preparation

The required quantities of PIB, terpene resin, paraffin wax, talcum powder, and BHT were weighed and combined according to Table I. The mixture was heated at 170–200 °C for about 12 h, stirring occasionally to ensure uniformity. Vegetable oil was then slowly added in and mixed after the main heating time. This was then heated for about 10 min and left to cool to room temperature. The same processing conditions were applied to all three formulations. The adhesive-gel formulations based on PIB (F1–F3) thus obtained are illustrated in Fig. 1.

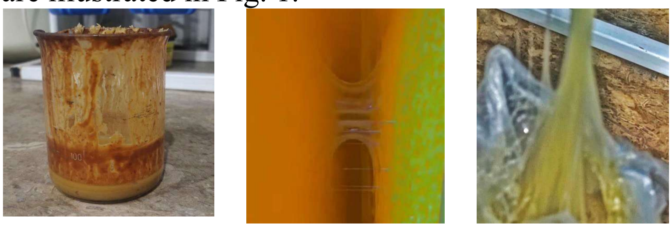

Fig. 1. Images Showing Three PIB-Based Rodent Adhesive-Gel Formulations (F1–F3).

C. Characterization

The formulations were tested in terms of adhesive activity, shear resistance and viscosity.

Adhesive strength assessment: Standardized cardboard or plastic substrates were used for the samples, which were conditioned at room temperature for 24 h. The adhesive behavior was evaluated in terms of peel testing at 180° configuration at a constant separation rate. Adhesion was not quantified in terms of peel force and was therefore reported as a comparative observation.

Shear-failure test: A fixed quantity of glue was used between fixed substrates with a fixed overlap area. The bonded samples were then conditioned at room temperature for about 24 h and then loaded with a constant load of 1 kg. Shear-failure time was recorded in minutes as the time for bond failure.

Viscosity measurement: The available viscometric method was used to measure the viscosity at 25 °C to compare the flow characteristics and consistency of F1–F3. No further instrumental specifications nor statistical treatment were added as there were no details of instrument or replicate given.

III. RESULTS AND DISCUSSION

A. Adhesive Performance

The 3 formulations exhibited different adhesive behaviour.

F1 (no terpene resin) showed the least adhesive performance. Observed adhesive behavior is improved with the incorporation of terpene resin in F2 and F3 and the highest recorded tackiness/adhesive performance is observed for F3.

This finding follows the trend of other tackifying components in pressure sensitive adhesive systems that can alter the nature of the interfacial contact and the ratio of adhesive to cohesive properties within a system. The research work carried out recently showed that the adhesive performance of PIB changes depending on its composition, and also that the adhesive performance of systems containing terpenes changes. [21-25]

Since this data set is not numerical peel force data, the result of the adhesion must be understood as a comparative performance observation rather than a statistical comparison of the adhesion - improvement.

B. Shear-Failure Behavior

Shear failure times of the three adhesive gel formulations based on PIB all increased as the terpene-resin content increased. The failure times of F1, F2 and F3 were 30, 120 and 150 min, respectively (Table II) indicating the shortest failure time for F1 followed by failure of F2 and F3 being the longest. So F2 had 4 times as long to fail in shear as F1 and F3 had 5 times as long. Under the loading conditions specified, therefore, the resistance to failure was highest for F3.

The failure time increase from F2 to F3 was relatively small; being about 25% higher. The results showed that the terpene resin incorporation level was correlated to the resistance to sustained loading within the range of formulations studied. The data available, however, do not support a determination of whether these trends would persist at increased concentrations of terpene-resin.

TABLE II. SHEAR FAILURE TIME AND VISCOSITY OF THE THREE PIB ADHESIVE-GEL FORMULATIONS.

Property	F1	F2	F3
Shear-Failure time (min)	30	120	150
Viscosity (Pa·s)	1.0	1.5	1.4
Relative Shear Performance	Lowest	Intermediate	Highest

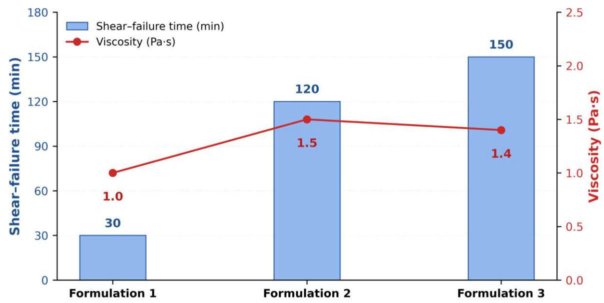

Fig. 2. Time to Shear Failure for the Three Adhesive-Gel Formulations Based on PIB under the Given Loading Condition.

C. Viscosity Behavior

The measured viscosity of F1, F2 and F3 was 1.0, 1.5 and 1.4 Pa·s, respectively (Table II). The highest viscosity was obtained for the F2 while the highest shear failure time was obtained for the F3 (Fig. 2).

This difference shows that the viscosity was not the only factor that determined resistance to sustained loading for the different adhesive formulations. The viscosity was the highest in F2, however the shear failure resistance was highest in F3. Thus, the adhesive performance of the formulations studied seemed to be dependent on the formulation composition and the resulting adhesive and rheological properties, but not on viscosity itself. [26-30]

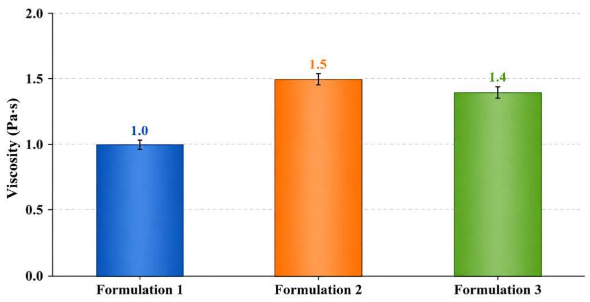

Fig. 3. Viscosity of the Three Adhesive-Gel Formulations Based on PIB at 25 °C.

D. Composition–Performance Relationship

The results show that the relative quantities of PIB and terpene resin influenced adhesive system performance.

F1 had the maximum amount of PIB and no terpene resin, whereas the amount of terpene resin gradually increased and the amount of PIB gradually decreased along F2 and F3.

The trend of the performance was: F1 → F2 → F3 for shear-failure resistance, with values of: 30 → 120 → 150 min.

But, there was an abnormality in viscosity: 1.0 → 1.5 → 1.4 Pa·s.

It is evident from the various trends that shear retention and viscosity are not synonymous characteristics. A formulation may have fairly high viscosity, but may not result in the highest shear failure resistance. [31-35]

This distinction demonstrates once again that the formulation performance is not solely based on the performance of a single measurable property, but rather depends upon the combined effect of the polymer matrix, tackifying component, filler system and other additives. [36-40]

E. Best Performing Formulation and Practical Significance

The overall performance of the three formulations was the best for F3. It had the longest shear-failure time (150 min) and the best qualitative adhesive/tackiness performance, while having a viscosity of 1.4 Pa·s, which is slightly lower than that of F2 (1.5 Pa·s). The results suggest that the highest viscosity was not associated with the highest resistance to shear failure, demonstrating the need to consider the formulation composition in determining the adhesive's performance.

It can be concluded, that in the range of formulations investigated F3 was the optimal formulation based on the measured properties. Its adhesive performance, shear resistance and viscosity are suitable and it has potential to be further developed. [41-43] The published laboratory measurements do not, however, provide any indication of the actual effectiveness of rodent capture and any application-specific and field testing would be necessary for further validation.

F. Cost Evaluation

The cost of all the raw materials was 49.05 PKR per unit of adhesive formulation (29.72 g). Total production cost 66.01 PKR/pad, which consisted of 15.00 PKR for packaging and labelling.

TABLE III. COST OF ADHESIVE-GEL FORMULATION AT LABORATORY LEVEL.

Raw Materials	Cost Details	Total Cost (PKR)
PIB (g)	26.4	23.49
Terpene Resin (g)	0.8	0.64
Paraffin Wax (g)	0.8	0.24
Talcum Powder (g)	0.8	0.24
BHT (g)	0.08	24.00
Vegetable Oil (g)	0.8	2.40
Total Raw Materials Cost (g)	29.72	51.01
Packaging and Labelling	Pad and Label Printing	15.00
Total Production Cost		66.01

This is an estimate of the cost of materials, packaging and labelling at the laboratory level.

IV. CONSTRAINTS AND FUTURE PROSPECTS

A. Limitations

Only three PIB–terpene-resin formulations were studied, however, and this prevented the formulation of a complete composition–property relationship. Peel strength was not quantifiable, and was therefore measured comparatively using adhesive performance. Other longer-term or advanced rheological properties, temperature and humidity stability and storage performance were not tested. Also, there was not enough replicate data that could be used for statistical significance testing. The costing only included material, packaging and labelling costs and was not a comprehensive techno-economic study.

B. Future Perspectives

Future studies are to be conducted with a more wide-spread variety of PIB–terpene–resin formulations through statistically designed formulation techniques. Peel strength and tack, dynamic rheology, relaxation behavior, viscosity as a function of temperature, environmental stability, aging, shelf-life and adhesion to substrates should be determined. [44-47] Additionally, controlled application-specific and field-performance studies must be carried out to ensure the practical usefulness and longevity of the developed adhesive system.

V. CONCLUSION

Three PIB-based adhesive-gel formulations containing different PIB content with various amounts of terpene-resins were formulated and tested with promising results with regard to their adhesive properties, shear resistance and viscosity. The results showed that the incorporation of terpene resin greatly enhanced the performance, as the shear failure time increased from 30 min in F1 to 120 min in F2 and 150 min in F3. While the highest viscosity (1.5 Pa·s) was found in F2, the highest shear-failure resistance (150 min) and the highest qualitative adhesive performance were obtained in F3 with a slightly lower viscosity of 1.4 Pa·s. These results show that the performance of the adhesive is dependent on the formulation composition and its adhesive/rheological behaviour and not just on viscosity. For the whole range of formulations investigated, best overall performance was achieved with F3. The use of wider formulation ranges, quantitative adhesion testing, advanced formulation rheological characterization, and environmental stability evaluations are needed to improve this formulation's applicability more broadly.

VI. ACKNOWLEDGMENT

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