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Optimisation of a Flame-Retardant PVC Plastisol Cover Compound for Conveyor Belts: Effect of Phosphate Ester Plasticiser, Chlorinated Paraffin Wax, Aluminium Trihydrate, and Zinc Borate on Flammability, Mechanical Properties, and Viscosity Stability

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Abstract

Flame-retardant PVC plastisol cover compounds for conveyor belts must balance fire safety, mechanical strength, and processability — properties that often work against each other. This study reformulated an industrial PVC plastisol cover paste by varying TCP, chlorinated paraffin wax (CPW), aluminium trihydrate (ATH), zinc borate, and DOP across five compositions (I–V), benchmarked against a baseline formulation (LOI 31%, abrasion loss 313 mm³, tensile strength 372 PSI, elongation 116%, Shore A 60). Formulations were evaluated for cost, solids content, LOI, DIN abrasion loss, tensile strength, elongation, hardness, and Brookfield viscosity (initial and after 7 days). Formulation IV showed the greatest mechanical improvement — abrasion loss dropped to 109 mm³, tensile strength rose to 1400 PSI, and elongation reached 220%, with LOI holding at 29% — but its viscosity increased more sharply during storage (40 to 78 Poise). Formulation V offered better viscosity stability (35 to 66 Poise) with LOI of 28.8% and abrasion loss of 117.9 mm³, though with a lower tensile strength of 1107 PSI. Overall, reducing total filler loading while redistributing phosphate ester plasticiser and zinc borate content helped reconcile flame retardancy with mechanical performance, while higher CPW and ATH levels tended to raise viscosity and reduce storage stability. Formulations IV and V stand out as the most promising candidates for scale-up, offering complementary strengths in mechanical performance and processing stability, respectively.

Keywords: *PVC plastisol; conveyor belt cover compound; flame retardancy; limiting oxygen index; chlorinated paraffin wax; zinc borate; aluminium trihydrate*

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1 INTRODUCTION

Polyvinyl chloride (PVC) plastisol cover compounds are used extensively on conveyor belts for underground coal mining and other hazard-classified duty because, unlike rubber covers, they can be formulated to be inherently flame- and heat-resistant while retaining good abrasion resistance and flexibility [1]. In these applications the cover compound must simultaneously satisfy at least three, often competing, requirements: (i) a limiting oxygen index (LOI) and flame-propagation performance sufficient to meet mine-safety and fire-hazard classifications; (ii) mechanical properties, principally abrasion resistance, ply/carcass tensile adhesion, elongation at break, and hardness, adequate for the mechanical duty of the belt; and (iii) a plastisol paste Viscosity that remains within a processable window, both immediately after mixing and after several days of storage, so that the paste can be coated and gelled without excessive thickening or sedimentation [2].

Flame retardancy in flexible PVC compounds is most commonly achieved using organophosphate ester plasticisers, of which tricresyl phosphate (TCP) and related aryl phosphates are the most established: in addition to plasticising the PVC matrix, they decompose on heating to release phosphorus-containing species that scavenge combustion radicals in the gas phase and promote protective char formation in the condensed phase [3]. Chlorinated paraffin wax (CPW) is widely used alongside phosphate esters as a lower-cost secondary plasticiser; because PVC itself already contains a high proportion of chlorine, the additional chlorine supplied by CPW further increases char formation and HCl release during combustion, contributing to flame retardancy at a lower cost than using phosphate ester alone, although at higher loadings CPW can compromise heat stability and exude from the compound [4]. Metal hydrate and borate fillers provide a complementary, largely condensed-phase route to flame retardancy: aluminium trihydrate (ATH) decomposes endothermically above approximately 180 °C, releasing water vapour that dilutes combustible gases and cools the substrate, while zinc borate acts synergistically with ATH, promoting char formation and suppressing smoke density; the combination of zinc borate and ATH has been reported to raise the LOI of rigid PVC substantially above that achieved by either filler alone [5,6]. Zinc borate is also used as a partial replacement for antimony trioxide in halogenated systems because it is comparatively low-cost and contributes strongly to condensed-phase char formation [7].

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A recurring difficulty in formulating flame-retardant PVC plastisols is that most of these flame-retardant strategies also change the rheology and mechanical behaviour of the compound. Phosphate ester and chlorinated paraffin plasticisers influence gelation kinetics and paste viscosity, while particulate fillers such as ATH and zinc borate raise paste viscosity roughly in proportion to their loading and surface area, and can promote thixotropic thickening on storage [2,8]. Reducing total filler and secondary-plasticiser loading to control Viscosity, however, typically comes at the expense of flame retardancy and can also reduce tensile strength and abrasion resistance if the primary plasticiser-to-resin ratio is not compensated. Because PVC plastisol is processed as a liquid dispersion rather than a melt, filler-filler and filler-plasticiser interactions that develop during storage are particularly consequential for cover-paste applications, since a paste that thickens excessively between mixing and coating cannot be used [9].

Most published work on flame-retardant PVC has examined individual additive classes in isolation-phosphate ester plasticisers [3], chlorinated paraffins [4], or ATH/zinc borate systems [5-7]-typically in rigid or generically plasticised PVC rather than in an industrial plastisol cover-paste matrix and rarely reports paste Viscosity stability over storage alongside flame and mechanical data. The combined effect of varying phosphate ester plasticiser, CPW, ATH, and zinc borate loading simultaneously, and benchmarking the resulting trade-offs against an industrial cover-paste specification, has not been systematically reported for conveyor belt applications.

2 MATERIALS AND METHODS

2.1 Materials

Emulsion-grade PVC resin (K-value grade used for plastisol coating) was used as the base polymer at 100 Phr in all formulations. Tricresyl phosphate/aryl phosphate ester (TCP) was used as the primary flame-retardant plasticiser. Chlorinated paraffin wax (CPW, 45% chlorine content) was used as a secondary plasticiser and auxiliary flame retardant. Dioctyl phthalate (DOP) was used as a supplementary primary plasticiser. Aluminium trihydrate (ATH) and a zinc borate dispersion were used as condensed-phase flame-retardant/smoke-suppressant fillers, together with a small addition of calcium carbonate ($CaCO_3$) in the baseline formulation only. An epoxidised plasticiser (epoxy plast) was used as a co-stabiliser/plasticiser, a carbon black dispersion was used for colouration, an antistatic agent was included to control surface

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resistivity, and a barium/zinc (Ba/Zn) heat stabiliser was used to protect against thermal dehydrochlorination during gelation.

2.2 Formulation Design and Preparation of PVC Plastisol

A present production baseline formulation and five reformulations (I, II, III, IV, and V) were designed by varying the phosphate ester plasticiser, CPW, ATH, DOP, and zinc borate loadings while holding the PVC resin, antistatic agent, epoxy plasticiser, carbon black dispersion, and Ba/Zn stabiliser levels constant. The overall strategy was to progressively reduce total compound loading (from 267.0 Phr in the baseline to between 168.5 and 238.5 Phr in the formulations) while redistributing the phosphate ester plasticiser-to-secondary-plasticiser ratio and the ATH-to-zinc-borate ratio, to determine whether a leaner, more efficiently balanced formulation could match or exceed the flame-retardant and mechanical performance of the baseline while remaining within the target processing Viscosity. The complete formulations, expressed in parts per hundred resins (Phr), are given in Table 1.

Table 1: PVC plastisol cover-paste formulations (Phr) - present baseline and five reformulations.

Ingredient (Phr)	Baseline	I	II	III	IV	V
PVC resin	100	100	100	100	100	100
TCP	40.0	55.0	55.0	30.0	37.5	33.5
CPW (45% Cl)	32.5	20.0	-	15.0	15.0	17.5
Antistatic agent	5.0	5.0	5.0	5.0	5.0	5.0
Epoxy plasticizer	2.0	2.0	2.0	2.0	2.0	2.0
ATH	60.0	40.0	20.0	-	10.0	10.0
$CaCO_3$	2.5	-	-	-	-	-
DOP	15.0	-	2.5	-	2.5	2.0
Carbon black dispersion	2.5	4.0	4.0	4.0	4.0	4.0
Zinc borate dispersion	5.0	10.0	20.0	10.0	10.0	11.0
Ba/Zn stabilizer	2.5	2.5	2.5	2.5	2.5	2.5
Total (Phr)	**267.0**	**238.5**	**211.0**	**168.5**	**188.5**	**187.5**

PVC plastisol formulations were prepared in the laboratory using a high-speed mixer at a speed of 1000 rpm for 30 min at room temperature (25 ± 2°C). The viscosity of the prepared paste was measured using a Brookfield viscometer at 12 rpm and a temperature of 25 ± 2°C.

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2.3 Sample Preparation

The prepared plastisol paste was placed into moulds of the required size and thickness. The samples were then heated in an oven for gelation and fusion at a controlled temperature of 180°C for 10–15 min. After heating, the samples were cooled to room temperature and removed from the mould. Care was taken to maintain a uniform thickness and smooth surface of the samples for accurate testing. Tensile, abrasion, and hardness measurements were performed on test specimens prepared according to the relevant ASTM standards.

Figure 1: Sample Preparation

2.4 Characterisation

Solids and liquids content (%) were determined gravimetrically from the non-volatile filler/resin fraction versus the plasticiser/liquid fraction of the paste. Flame retardancy was assessed by the limiting oxygen index (LOI), the minimum oxygen concentration in an oxygen-nitrogen mixture required to sustain candle-like combustion of the gelled sheet, consistent with the LOI methodology widely used to benchmark flame-retardant PVC formulations [5-7]. Abrasion resistance was measured as volume loss (mm^3) on a DIN-type rotating-drum abrasion tester according to ISO 4649, following the abrasion-loss convention used for conveyor belt cover-compound grading [10]. Tensile strength was measured as the cover-to-carcass ply adhesion / breaking strength in the weft direction (denoted PSI) using the manufacturer's internal tensile test method, consistent in principle with peel/breaking-strength tests used for

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coated fabric and belt cover qualification. Elongation at break (%) was measured on the same tensile specimens. Hardness was measured using a Shore A hardness tester according to ASTM D2240. Paste Viscosity was measured on a Brookfield Viscometer spindle no.12 at a fixed spindle speed and temperature, both immediately after mixing (initial) and after seven days of ambient storage, to capture thixotropic build-up and filler-plasticiser interaction effects on shelf stability.

3 RESULTS AND DISCUSSION

Table 2: Measured properties of the present baseline and five formulations against the target specification.

Property	Baseline	I	II	III	IV	V
LOI (%)	31.0	33.0	29.0	30.0	29.0	28.8
Abrasion loss (mm^3)	313.0	251.0	235.0	203.0	109.0	117.9
Tensile strength (PSI)	372.0	993.0	1085.0	1040.0	1400.0	1107.0
Elongation at break (%)	116.0	266.0	238.0	145.0	220.0	184.0
Shore A hardness	60.0	65.0	62.0	70.0	70.0	72.0
Viscosity, initial (Poise)	37.0	36.0	37.0	68.0	40.0	35.0
Viscosity, 7 days (Poise)	61.0	55.0	55.0	122.0	78.0	66.0

3.1 Flame Retardancy (LOI)

The baseline formulation achieved an LOI of 31%, the highest of all six formulations, reflecting its high combined loading of ATH (60 Phr) and TCP (40 Phr). I, which raised the phosphate ester plasticiser to 55 Phr while retaining 40 Phr ATH, achieved the highest LOI among the reformulated candidates (33%), consistent with the combined gas-phase (phosphorus) and condensed-phase (ATH-dehydration) flame-retardant mechanisms acting together [3,5]. Reducing ATH loading to 20 Phr or below while increasing zinc borate (II, III, IV, V) reduced LOI to between 28.8 and 30%, still above the 28% target specification in every case. This is consistent with reports that zinc borate and ATH act synergistically rather than additively, such that a given LOI can be maintained with a lower total filler loading provided the zinc borate fraction is increased as ATH is reduced [5,6]. V, with the lowest total ATH + zinc borate loading among the low-filler candidates (21 Phr) but the highest zinc borate-to-ATH ratio

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(1.1:1), achieved an LOI of 28.8%, only marginally above the target and the lowest of the six formulations, indicating that further reduction in total filler loading without increasing the zinc borate fraction proportionally would risk falling below the flame-retardancy specification.

3.2 Abrasion Resistance

Abrasion loss decreased substantially and near-monotonically as total filler/plasticiser loading was reduced and rebalanced, from 313 mm^3 in the baseline to a minimum of 109 mm^3 in IV, a 65% reduction. This improvement is attributed principally to the lower ATH loading (10 Phr in IV versus 60 Phr in the baseline): coarse, high-loading mineral filler such as ATH is known to create stress-concentration sites at the filler-matrix interface, and its debonding under abrasive shear accelerates material loss, whereas a leaner filler system with a higher proportion of plasticiser allows the PVC matrix to absorb abrasive energy more effectively. V achieved a similarly low abrasion loss (117.9 mm^3), indicating that this benefit is not unique to IV but is a general consequence of the reduced ATH loading strategy used across III, IV, and V (all at or below 15 Phr CPW and 10 Phr ATH). II, which used the highest zinc borate loading (20 Phr) but no CPW, showed an intermediate abrasion loss (235 mm^3), suggesting that the finer zinc borate particles are somewhat less abrasive than the coarser ATH, but that CPW also contributes to abrasion resistance, likely through its plasticising effect on matrix flexibility.

3.3 Tensile Strength (PSI) and Elongation at Break

The baseline formulation exhibited by far the lowest tensile strength (372, PSI) and elongation at break (116%) of the six formulations, falling well short of the 1200 (PSI) and 153% targets, respectively. This is consistent with the very high total filler/plasticiser loading (267 Phr) in the baseline, diluting the PVC matrix and creating a stiffer, more heavily filled compound that limits chain mobility and load transfer during tensile deformation. All five reformulations more than doubled the tensile strength relative to baseline, with IV achieving the highest value (1400, PSI), exceeding the target by 17%, and II achieving the second highest (1085). Elongation at break followed a broadly similar trend, with I achieving the highest value (266%) and III the lowest among the reformulated candidates (145%, just below the 153% target). III combined the lowest CPW-plus-ATH loading with the lowest DOP content of the reformulated candidates, which appears to have restricted chain mobility during elongation despite its relatively favourable abrasion and cost performance, illustrating that reducing plasticiser loading too aggressively can compromise elongation even when tensile strength remains acceptable. IV's combination of moderate CPW (15 Phr), moderate DOP (2.5 Phr), and a

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balanced ATH/zinc borate ratio (10:10 Phr) appears to provide the most favourable balance of plasticisation and reinforcement for simultaneous tensile strength and elongation performance.

3.4 Hardness

Shore A hardness increased from 60 in the baseline to between 62 and 72 across the formulations, with IV and III both meeting the 70 targets exactly and V exceeding it slightly (72). The lower hardness of the baseline, despite its very high ATH filler loading, is consistent with its correspondingly higher total plasticiser content (TCP plus CPW plus DOP totalling 87.5 Phr), which softens the compound; in the reformulated candidates, the combination of reduced total plasticiser loading and a rebalanced ATH/zinc borate filler system raised hardness towards, and in the case of V slightly beyond, the target specification.

3.5 Viscosity and Storage Stability

Viscosity was the property most sensitive to the reformulation strategy and represents the principal trade-off identified in this study. The baseline paste viscosity (37 Poise initially, 61 Poise after seven days) already marginally exceeded both the 35 Poise and 60 Poise targets. I and II, which retained a relatively high phosphate ester plasticiser loading (55 Phr) together with either moderate CPW (20 Phr) or no CPW, showed the best viscosity stability of all formulations (36/55 and 37/55 Poise, respectively), indicating that phosphate ester plasticiser at this loading level does not itself drive excessive thixotropic build-up. In contrast, III, which combined a comparatively low phosphate ester loading (30 Phr) with the total absence of ATH, showed a pronounced and desirable Viscosity increase (68 Poise initial, rising to 122 Poise after seven days, more than double the target), suggesting that removing ATH entirely, rather than simply reducing its loading, disrupts the particle-packing behaviour of the paste and promotes flocculation or thixotropic gelation of the remaining zinc borate and carbon black dispersions during storage. IV, which reintroduced a moderate ATH loading (10 Phr) alongside the same zinc borate level as III, showed a substantially improved but still out-of-specification Viscosity profile (40/78 Poise), confirming that a minimum ATH loading of approximately 10 Phr helps stabilise paste rheology relative to zero ATH, though not sufficiently to meet the target at this total plasticiser loading. V, which used a marginally lower phosphate ester loading (33.5 Phr) and a marginally higher CPW loading (17.5 Phr) than IV, together with the same 10 Phr ATH but a slightly higher zinc borate content (11 Phr), achieved the closest match to the Viscosity specification of all reformulated candidates (35/66 Poise initial and seven-day,

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respectively), essentially meeting the initial-viscosity target and coming within 10% of the seven-day target.

Taken together, the viscosity data indicate that (i) ATH loading below approximately 10 Phr, and in particular its complete removal, is the dominant driver of unacceptable thixotropic thickening in this compound system, and (ii) once a minimum ATH loading is restored, fine adjustments to the phosphate ester plasticiser-to-CPW ratio (as in the difference between IV and V) provide a secondary but still meaningful lever for tuning storage Viscosity without materially changing flame retardancy.

3.6 Morphological Analysis (SEM):

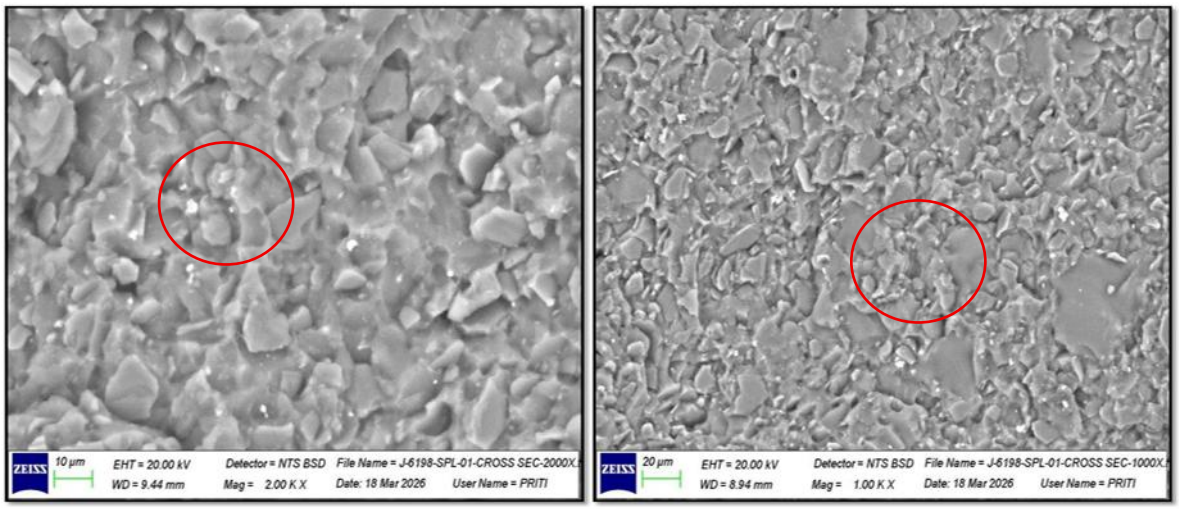

Figure 2: VI Sample Scanning Electron Microscopy Analysis

Scanning electron micrographs of the cross-section of the gelled Proposed VI cover-compound sheet are shown in Figure 1, acquired using a backscattered-electron detector at two magnifications (2000x, Figure 1a; 1000x, Figure 2) to examine filler dispersion and matrix-filler morphology at both local and larger-field scale.

At both magnifications, the filler phase (predominantly ATH and zinc borate, present at 10 and 11 Phr respectively in Proposed VI) appears as densely packed, angular to sub-angular particles ranging in size from below 1 um up to approximately 5-8 um, distributed with reasonable uniformity across the field of view and showing no large-scale agglomerate clusters at either magnification. This is consistent with the reduced total filler loading of Proposed VI (21 Phr ATH + zinc borate combined, versus 65 Phr in the baseline) allowing more effective wetting and dispersion of the filler particles by the plasticiser/resin matrix during mixing.

3.7 FTIR Spectrum with Baseline Compound

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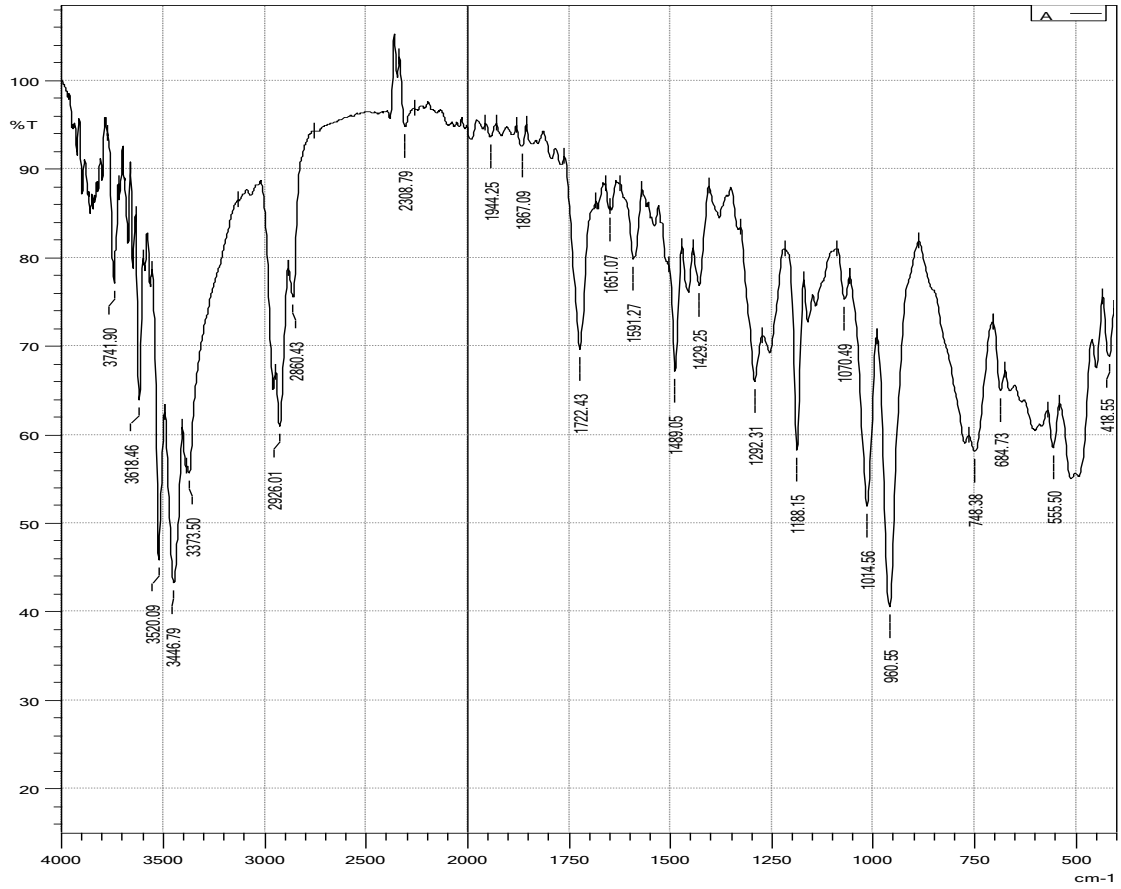

Figure 3: FTIR Spectrum with Baseline Compound

The baseline formulations have absorption bands that are typical of the plasticised PVC matrix. These results indicate that the base polymer structure of the PVC matrix stays the same when fillers are added. The main absorption band at 960 cm^{-1} is due to the C–Cl stretching vibration, which is a key feature of the PVC backbone of the plasticised PVC matrix. A strong band at 1722 cm^{-1} is from the plasticiser that's in all formulations of the plasticised PVC matrix. The C–H asymmetric and symmetric stretching vibrations of the CH_2 groups are seen at 2926 cm^{-1} and 2860 cm^{-1}, which is what we expect from the plasticiser's long aliphatic chain in the plasticised PVC matrix. The bands at wavenumbers like 748 cm^{-1}, 684 $cm^{-1}$and 555 cm^{-1} are from the C–H out-of-plane deformation and the skeletal vibrations of the PVC chain of the plasticised PVC matrix.

3.8 Overall Comparison Against Target Specification

Of the six formulations evaluated, none simultaneously met every element of the target specification. IV met or exceeded the LOI, abrasion, tensile strength, elongation, and hardness targets, and represents the strongest mechanical and flame-retardant candidate overall, but in viscosity (40/78 Poise) remained above specification, particularly after seven days of storage,

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which would need to be addressed, for example through a minor further increase in ATH or a rheology modifier, before it could be adopted in production without a shortened paste shelf life. V met or nearly met the LOI (28.8% versus 28% target), abrasion, hardness, and initial-viscosity targets, and came within 8% of the tensile strength target and within 10% of the seven-day Viscosity target, making it the most balanced single candidate across all seven performance criteria simultaneously, at a lower cost than IV (Rs 131.2/kg versus Rs 134.9/kg). The choice between IV and V in practice depends on whether mechanical performance or processing/shelf-life stability is prioritised for the intended duty of the belt.

4 CONCLUSION

This study systematically reformulated an industrial PVC plastisol cover-paste compound by varying phosphate ester plasticiser TCP, chlorinated paraffin wax, aluminium trihydrate, zinc borate, and DOP loading across five compositions relative to a production baseline, and evaluated the resulting trade-offs between flame retardancy, mechanical performance, and paste viscosity stability. Reducing total compound loading from 267 to between 169 and 239 Phr, while redistributing the ATH-to-zinc-borate ratio and moderating the phosphate ester and CPW content, more than tripled tensile strength and roughly doubled elongation at break relative to the baseline, while reducing abrasion loss by up to 65% and maintaining LOI above the 28% target in every formulation. Paste viscosity was the most difficult property to control: complete removal of ATH (III) caused a pronounced and unacceptable viscosity increase on storage, while restoring a moderate ATH loading of approximately 10 Phr (IV and V) substantially improved, but did not fully eliminate, this effect. IV offered the best overall mechanical and flame-retardant performance but exceeded the viscosity specification, while V offered the closest overall match to the full target specification, including viscosity, at a marginally lower cost, at the expense of a tensile strength below target. These findings indicate that a minimum threshold ATH loading (around 10 Phr in this system) is required to maintain acceptable paste rheology irrespective of zinc borate content, and that the phosphate ester-to-CPW ratio provides a further, finer lever for balancing flame retardancy, mechanical performance, and processability in PVC plastisol conveyor belt cover compounds. IV and V are recommended as the two most promising candidates for pilot-scale trial, representing performance-optimised and processing-optimised formulations, respectively.

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Author Contributions

[Author a]: Conceptualisation, Methodology, Investigation, Writing - Original Draft.

[Author b]: Supervision, Resources, Writing - Review & Editing.

Conflict of Interest

The authors declare no conflict of interest.

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