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RESEARCH ARTICLE

Development and characterization of linseed-mustard and castor-mustard oil coated DAP formulations

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Abstract

Oil based coatings provide a biodegradable and low-cost strategy for developing Controlled-Release Fertilizers (CRFs). In this study, 18 oil-coated phosphorus (P) fertilizer formulations were prepared using diammonium phosphate (DAP) as core nutrients and linseed, castor and mustard oils in defined blend ratios (100:0, 75:25, 50:50) at three coating levels (5 %, 10 %, 15 %). Curing behavior was assessed for 45 days, followed by characterization using Fourier Transform Infrared (FTIR) spectroscopy and Scanning Electron Microscopy (SEM). Phosphorus release kinetics were evaluated in an aqueous medium over 30 days and fitted to both first-order and Korsmeyer-Peppas (KP) models. Curing data indicated that 100 % linseed oil underwent the highest oxidation-driven weight gain (≈5 % by day 10) followed by shrinkage, whereas 50:50 oil blends showed minimal weight fluctuations (< 1 % by day 10) and stable film formation. FTIR confirmed oxidation and polymerization (C-O-C, C=O, C=C) with preservation of phosphate bands. SEM revealed smooth, continuous coatings for linseed oil-mustard oil (LM) formulations and thicker, more flexible films for castor oil-mustard oil (CM) formulation. Release studies showed significantly reduced dissolution from all coated fertilizers (60–70 % in case of LM coated DAP and 60–80 % in case of CM coated DAP) compared to uncoated DAP (~94 % release) at 30 days. Kinetic modeling demonstrated that most LM-coated DAP formulations followed Korsmeyer-Peppas (non-Fickian) behavior, whereas several CM formulations followed first-order kinetics. Overall, LM blends at 75:25 and 50:50 with a 10 % coating level exhibited optimal curing stability and controlled-release behavior, marking them promising formulations for subsequent soil incubation and greenhouse evaluation.

Keywords: controlled-release fertilizer; FTIR; Korsmeyer-Peppas; linseed oil; phosphorus release; SEM

Introduction

Phosphorus is an essential macronutrient required for energy transfer, cell division, root development and biosynthesis of nucleic acids and phospholipids. Despite its importance, the agronomic phosphorus use efficiency rarely exceeds 15–25 % in most soils due to precipitation, fixation by Fe- and Al-oxides in acidic soils and Ca-bound complexes in alkaline and calcareous soils (1, 2). As global demand for food rises, improving P-use efficiency has become a strategic priority for sustainable intensification of agriculture.

Controlled release fertilizers (CRF) provide a promising approach to synchronize nutrient release with plant demand, reduce nutrient losses, minimize fixation and increase fertilizer use efficiency (3, 4). Among the various CRF development strategies including polymer encapsulation, matrix entrapment, thermal modification and nano-enabled formulations, surface coating remains one of the simplest, most cost-effective and scalable approaches (5, 6). Coatings constructed from hydrophobic or semi-permeable materials create diffusion barriers that regulate water penetration and nutrient dissolution (7).

Bio-based oils have recently gained attention as sustainable coating materials because they are biodegradable, inexpensive and possess tunable chemical functionality (8, 9). Drying oils such as linseed polymerize through oxidative crosslinking to form rigid films, while castor oil develops flexible coatings due to the presence of hydroxylated fatty acids (10). Mustard oil, with its high monounsaturated fatty acid content, slows polymerization and prevents cracking of the coating (11). Therefore, blending drying and non-drying oils offers opportunities to tailor mechanical stability, hydrophobicity, polymerization kinetics and nutrient release behaviour.

Recent studies have demonstrated the potential of oil derivatives in nutrient encapsulation. For example, linseed oil based coatings on urea improved nitrogen retention (12). Polyurethane coatings synthesized from castor and soybean oils achieved extended nutrient release and reduced volatilization (13). Bio-oil derived from pyrolysis also enhanced longevity of coated fertilizers (14). Despite these advances, very limited work has been conducted on oil-coated phosphorus fertilizers, especially for DAP and no systematic comparison exists for blended oil coating using linseed, mustard and castor oils.

Thus, this work aims to develop oil-coated phosphorus fertilizer formulations and to characterize them using curing behavior, FTIR, SEM and water-based P release kinetics. The hypothesis of the current research is that coating blended oils may create physical barriers and help in slow release of nutrients from the coated fertilizers at optimum levels and the objective is to study the release behavior of the synthesized oil coated controlled release formulations.

Materials and Methods

The present laboratory experiment was conducted at the Division of Soil Science and Agricultural Chemistry, ICAR-Indian Agricultural Research Institute, New Delhi, India during 2022–23.

Preparation of oil-coated controlled release formulations

Commercially available linseed oil, mustard oil and castor oil purchased from local market were used for this experiment. The oils were blended on a magnetic stirrer-cum-heater at 300 rpm for 2 hr at 70 °C under open air condition so that the heating and oxidation will increase ionization of carboxyl groups (-COOH) and breaking of C=C double bonds and initiatefree-radical polymerization. Resulted boiled linseed oil became denser and darker in colour and is called double boiled linseed and was subsequently used for oil-based formulations. Six oil blends were prepared : 1) 100 % linseed oil (L_{100}), 2) 75:25 blended linseed oil + mustard oil ($L_{75}M_{25}$), 3) 50:50 blended linseed oil + mustard oil ($L_{50}M_{50}$), 4) 100 % castor oil (C_{100}), 5) 75:25 blended castor oil + mustard oil ($C_{75}M_{25}$) and 6) 50:50 blended castor oil + mustard oil ($C_{50}M_{50}$). Finally, a portion of each oil formulation was transferred into Petri dishes to periodically measure the percentage change in weight (W %), while the remaining portion was preserved in airtight containers for subsequent fertilizer coating. Commercial grade DAP granules were coated at 5 %, 10 % and 15 % (w/w) with these oils, air dried and stored in airtight containers resulting in 18 oil coated fertilizer formulations.

Study on curing behavior of oils

Oil formulations poured into Petri dish were monitored for weight change at regular intervals over 45 days and percent weight change W (%) was calculated as follows:

$$W\,(\%) = \frac{(W_t - W_i)}{Wi} \times 100 \qquad \text{(Eqn. 1)}$$

Where, W_i and W_t represents the initial weight of the formulation at day 0 and weight at time t.

FTIR spectroscopy

FTIR for initially finely powdered samples were recorded using the Bruker: ALPHA II, FTIR system. Samples were scanned between 4000–500 cm^{-1} IR spectra typically scanned at 4 cm^{-1} scanning resolution with 24 times continuous scanning. Peaks were assigned to oxidation, polymerization and phosphate functional groups.

SEM imaging

Surface and cross-sectional morphology of uncoated, LM-coated and CM-coated DAP were analysed through scanning using the SEM, Techno FEI in Department of Nanoscience and Technology, TNAU, Coimbatore. Samples were assessed after thin-layer coating of gold-palladium sputter coating.

Water release kinetic study of prepared oil-coated controlled release fertilizer formulations

Coated fertilizer formulations and uncoated DAP were incubated in distilled water at 30 °C. Aliquots collected at 0, 2, 4, 8, 12, 20 and 30 days were analyzed for phosphorus using the vanadomolybdo-phosphate method (15). Nutrient release kinetics were assessed through first-order model (Eqn. 2) and semi-empirical KP model (Eqn. 3).

$$\ln M_t = \ln M_0 - K_1 t \qquad \text{(Eqn. 2)}$$

$$\frac{M_t}{M_0} = K_m t^n \qquad \text{(Eqn. 3)}$$

Where, M_0 is the initial concentration of nutrient (P), M_t is the concentration of nutrients in solution at time t; K_1 is the first-order rate constant, K_m is the KP constant and *n* is the release exponent used as release mechanism indicator.

R^2, adjusted R^2 and Standard Error of Estimates (SEE) were used to determine best fit.

Results and Discussion

Study on curing behavior of oils

The 100 % linseed oil formulation exhibited the highest early weight gain, reaching its maximum around day 9 before gradually declining thereafter. The 75:25 LM blend also showed an initial rise in weight followed by a steady decrease after day 15. In contrast, the 50:50 LM formulation maintained highly stable weights with only minor fluctuations throughout the curing period. Among the castor-based formulations, 100 % castor oil displayed a continuous increase in weight over time, while the 75:25 CM blend showed moderate early weight gain before stabilizing. The 50:50 CM formulation remained the most stable of the castor blends, exhibiting minimal variation across the entire curing duration (Fig. 1). Percent weight change patterns closely reflected the raw weight data, with linseed-based formulations showing comparatively larger fluctuations and castor-based formulations demonstrating more consistent curing trends.

The curing patterns observed among the 6 oil formulations can be attributed to the inherent chemical behavior of drying and non-drying oils. The early weight gain followed by contraction in the 100 % linseed formulation is characteristic of strong oxidative polymerization, forming a dense crosslinked network as previously described for linseed-oil based films (16, 17). Incorporation of mustard oil moderated these fluctuations, with the 50:50 LM blend exhibiting highly stable curing, consistent with reports that monounsaturated oils reduce excessive crosslinking and improve film stability (11). In contrast, castor-based formulations showed gradual and sustained weight gain without shrinkage, attributable to the hydroxyl-rich ricinoleic acid backbone that promotes flexible film formation rather than classical oxidative drying, in agreement with polyurethane-type coatings derived from castor oil (9, 18). The 50:50 CM blend displayed the most stable behaviour among

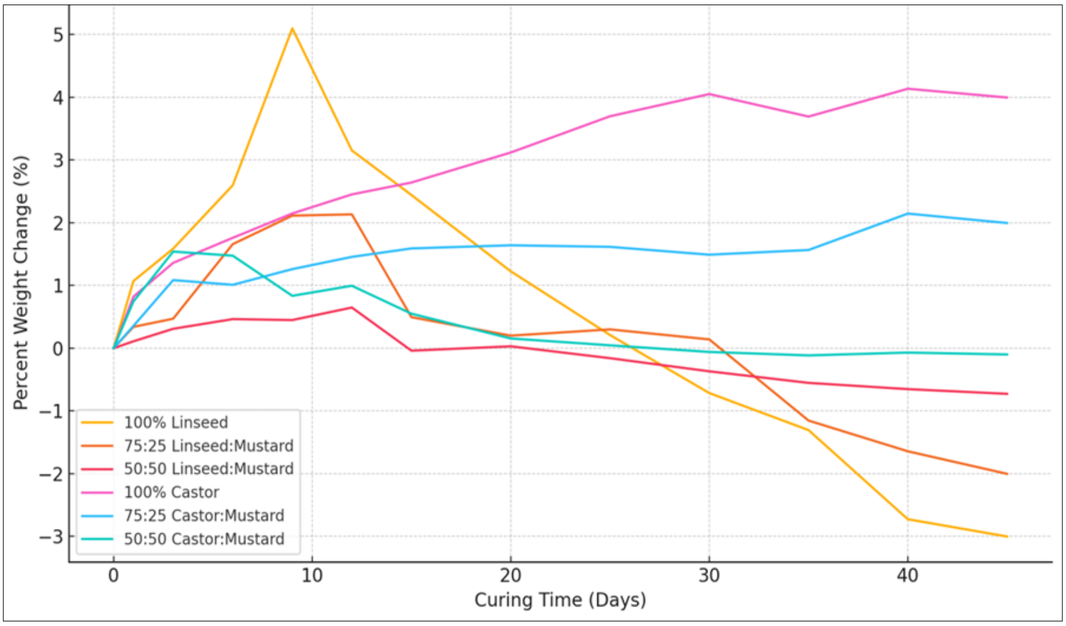

Fig. 1. Percent change in weight of oil formulations with time during curing.

castor formulations, supporting findings that mixed-oil systems provide balanced mechanical and oxidative properties for coating applications (8). Overall, the comparative curing behaviour highlights that 50:50 LM and 50:50 CM formulations offer the most stable films, making them promising candidates for creating durable coatings in CRFs, as stability during curing is closely linked to coating integrity and long-term release performance (3, 19).

FTIR functional group analysis

FTIR analysis clearly distinguished the chemical characteristics of uncoated and oil-coated DAP formulations, evidencing effective surface encapsulation and blended oil polymerization. The uncoated fertilizer (Fig. 2a) exhibited diagnostic phosphate vibrational bands at 910 cm^{-1} (P-O symmetric stretching), 1093 cm^{-1} (P-O asymmetric stretching) and 1165 cm^{-1} (P=O stretching), together with NH_4^+ stretching at 3061 cm^{-1}, representing the intrinsic spectral peaks of the DAP core. Upon coating, additional absorption features emerged that were attributed to oxidized oil films. LM coatings (Fig. 2b) showed pronounced bands at 1025 cm^{-1} (C-O stretching) and 1070 cm^{-1} (C-O-C linkages), indicative of peroxide formation and ester network development, along with intense carbonyl absorptions at 1715–1808 cm^{-1} associated with oxidative crosslinking of polyunsaturated fatty acids via classical auto-oxidation mechanisms (16, 17). The presence of residual C=C stretching near 1685 cm^{-1} and ester carbonyl peaks around 1740 cm^{-1} in the 50:50 LM formulation suggests partial oxidative propagation and formation of semi-rigid polymeric films characteristic of drying oils (11). In contrast, CM coatings (Fig. 2c) exhibited dominant O-H stretching at 3441 cm^{-1} and C-O absorptions at 1218 cm^{-1}, along with aliphatic C-H vibrations at 2922 cm^{-1}, reflecting hydrogen-bonded network formation driven by hydroxyl-functional ricinoleic acid and favouring flexible film architectures rather than extensive oxidative crosslinking, consistent with reports on castor oil based polyurethane coatings (9, 10). Notably, phosphate vibrational bands were retained across all coated samples, confirming that the oil films function as physical encapsulating layers without chemically interacting with the fertilizer constituents, in agreement with prior controlled release fertilizer coating studies (8, 19). Overall, these FTIR results demonstrated composition-dependent polymerization pathways, effective film formation and chemical stability of the DAP core under oil-coating conditions.

SEM analysis

SEM micrographs of uncoated (Fig. 3a–c) and oil-coated DAP granules (Fig. 3d–i) revealed pronounced morphological modifications induced by film formation. Uncoated DAP exhibited irregular crystalline surfaces with sharp edges, exposed micropores and loosely bound particles, consistent with the inherent morphology of conventional DAP. In contrast, LM coated granules showed markedly smoother external surfaces even at low magnification (80 ×), indicating uniform spreading of the oil film, while higher magnifications (500 × and 2000 ×) revealed partial masking of crystal facets and infilling of surface voids, suggesting effective surface encapsulation. Cross-sectional imaging confirmed the presence of a thin, continuous LM coating adhering closely to the fertilizer core, consistent with oxidative curing of linseed oil that promotes uniform film formation through crosslinked network development (16, 17) and pore sealing, as previously reported for drying-oil-coated fertilizers (8). In comparison, CM coated granules exhibited thicker, more cohesive coatings with rounded and compact surface morphology and high-magnification images showed a dense, homogeneous polymer layer. Cross-sections further revealed a clearly distinguishable, flexible coating enveloping the granule, reflecting the hydroxyl-rich ricinoleic acid structure of castor oil that favors elastic, hydrogen-bonded polymer networks rather than rigid oxidative films (18). Similar dense and continuous morphologies have been reported for castor oil derived polyurethane fertilizer coatings, where enhanced elasticity improves resistance to cracking during handling and dissolution (9). Overall, these SEM observations demonstrate that oil composition strongly governs coating thickness, texture, integrity and structural stability of CM coatings indicating greater potential for sustained nutrient release and improved fertilizer performance, in agreement with earlier reports on biopolymer-based controlled-release systems (3, 19).

Study on water release kinetics and model fitting

The water release kinetics of coated and uncoated DAP fertilizers

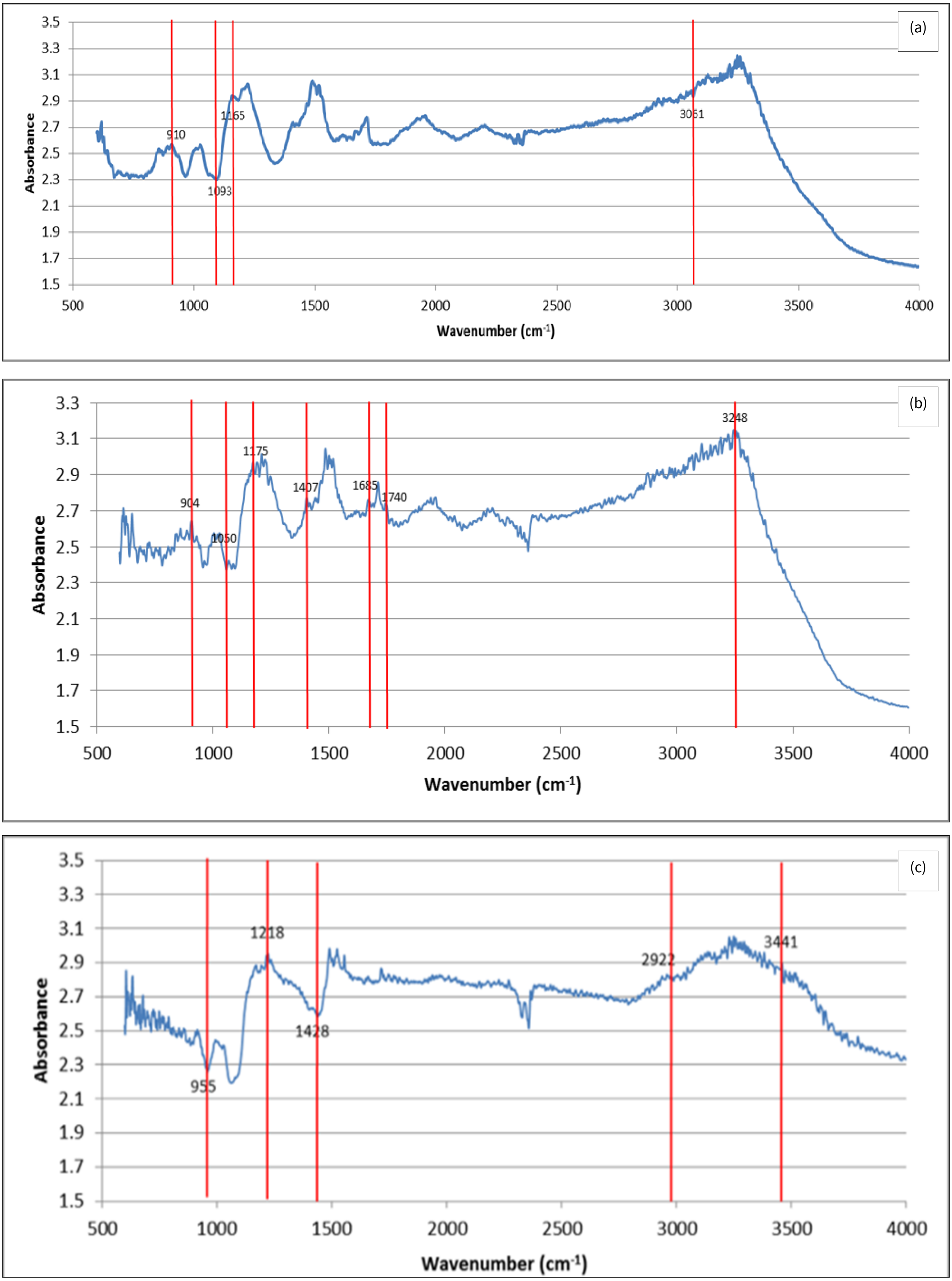

Fig. 2. FTIR spectra of uncoated and oil-coated DAP.

a) uncoated DAP, b) linseed oil-mustard oil coated DAP, c) castor oil-mustard oil coated DAP

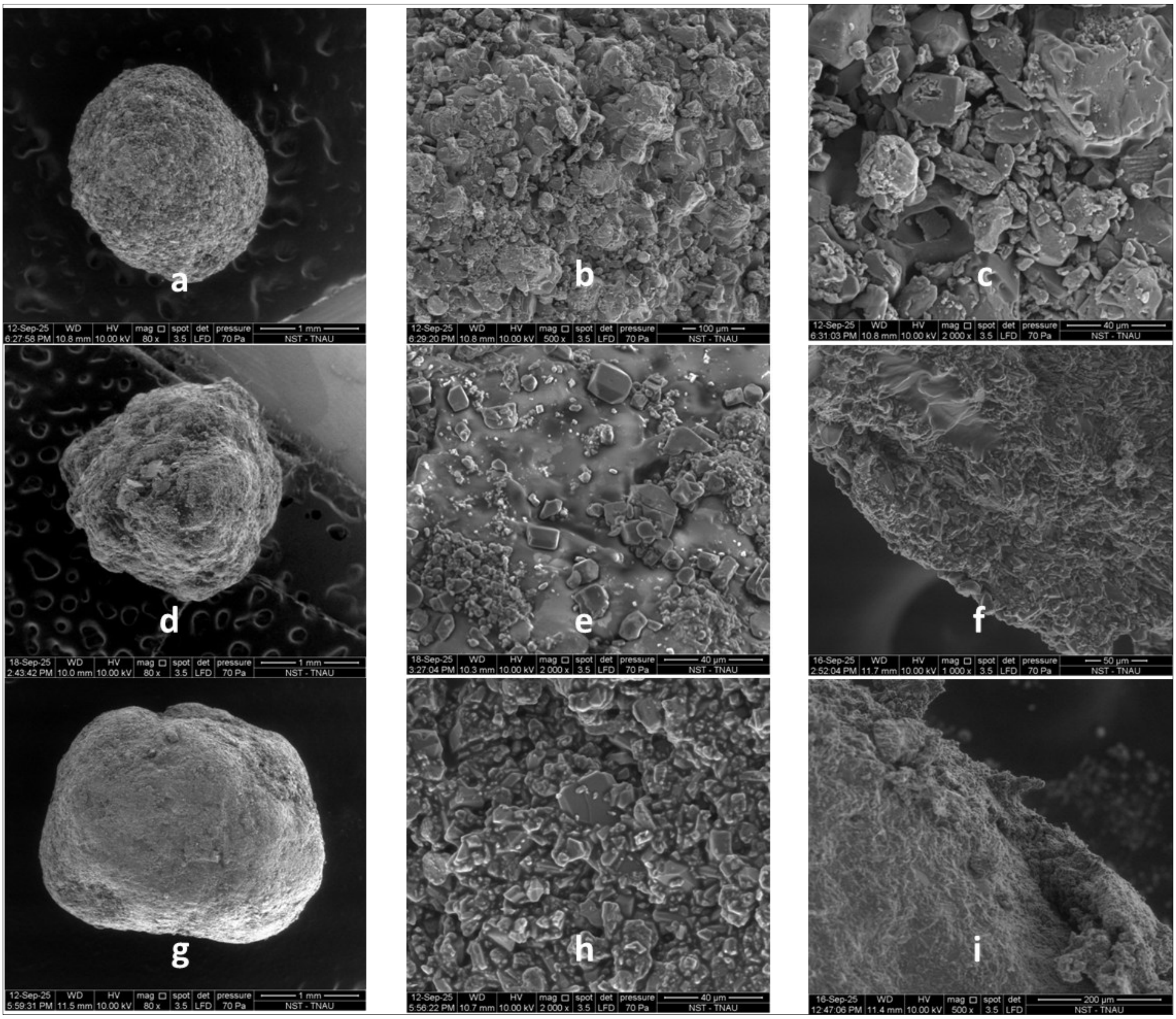

Fig. 3. SEM images of uncoated and oil-coated DAP. (a) 80 x on surface of uncoated DAP, (b) 500 x on surface of uncoated DAP, (c) 2000 x on surface of uncoated DAP, (d) 80 x on surface of LM coated DAP, (e) 2000 x on surface LM coated DAP, (f) Cross section showing LM layer over DAP, (g) 80 x on surface of CM coated DAP, (h) 2000 x on surface of CM coated DAP, (i) Cross section showing CM layer over DAP

exhibited clear differences in dissolution behaviour over 30-days incubation period. Uncoated DAP showed rapid solubilization, releasing approximately 18 % phosphorus at day 0, 56.18 % by day 2 and over 93 % by day 30, reflecting its inherently high-water solubility. In contrast, LM coated formulations exhibited substantially retarded phosphorus release, with cumulative dissolution limited to 60–69 % by day 30 depending on oil blend and coating level (Fig. 4a) and higher coating thicknesses (10 and 15 %) consistently suppressing early-stage release relative to 5 % coatings. CM coated formulations showed greater variability in release behaviour, with cumulative phosphorus dissolution ranging from 70–82 % by day 30 in treatments with higher castor oil content, while certain formulations such as CM 75:25 (5 %) exhibited markedly reduced release due to formation of cohesive, diffusion-limiting films (Fig. 4b). These trends can be attributed to the hydrophobic barrier imparted by the oil coatings, which restricts water penetration and delays dissolution of the fertilizer core; in particular, the strong early-release suppression observed in LM-coated DAP is consistent with oxidative polymerization of drying oils forming semi-permeable networks that limit rapid solubilization (8). The release profiles of CM coatings align with reports that castor-oil-derived polymers generate thicker, flexible films governing nutrient diffusion rather than dissolution-controlled release (9, 10). Overall, the characteristic reduction in initial phosphorus release followed by gradual nutrient release underscores the effectiveness of oil-based coatings in modulating dissolution kinetics, supporting their potential to enhance phosphorus-use efficiency and sustained nutrient availability in agricultural systems (3, 7).

Model fitting of phosphorus release data revealed distinct kinetic behaviours among coated and uncoated DAP formulations. Most LM coated DAP granules exhibited conformity with the KP model, as indicated by higher R^2 and adjusted R^2 values and lower SEE. It suggested anomalous (non-Fickian) release governed by the combined effects of nutrient diffusion and coating relaxation or swelling (Table 1). In contrast, several CM coated formulations, particularly those containing higher proportions of castor oil, were better described by the first-order model, indicating predominantly diffusion-controlled release. The preference of the KP model for LM-coated formulations is consistent with the presence of oxidized functional groups and

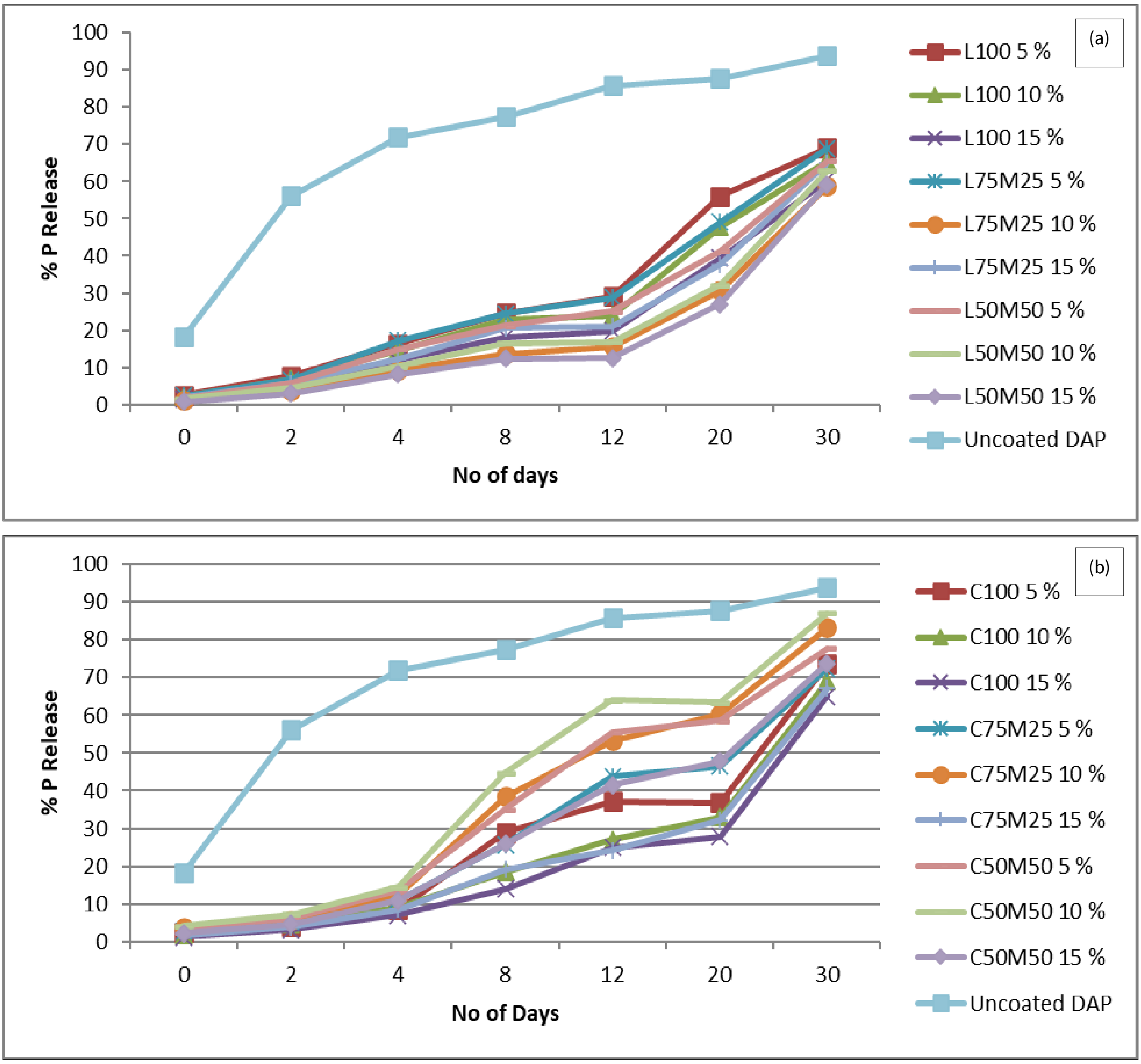

Fig. 4. Percent phosphorus release from different oil-combination coated diammonium phosphate formulations. (a) linseed oil + mustard oil coated DAP formulations, (b) castor oil + mustard oil coated DAP formulations in water medium

Table 1. Model fitting based on water release kinetics for different oil coated DAP fertilizer formulations

Treatment	First-order kinetic model			Korsemeyer-Peppas (KP) model			Best model
	R2	Adj R2	SEE	R^2	Adj R^2	SEE	
L_{100} 5 %	0.982	0.973	3.631	0.983	0.974	3.543	KP
L_{100} 10 %	0.979	0.968	3.646	0.982	0.973	3.342	KP
L_{100} 15 %	0.983	0.975	2.945	0.984	0.977	2.848	KP
$L_{75}M_{25}$ 5 %	0.984	0.975	3.312	0.989	0.983	2.717	KP
$L_{75}M_{25}$ 10 %	0.942	0.914	5.572	0.974	0.960	3.775	KP
$L_{75}M_{25}$ 15 %	0.976	0.964	3.520	0.978	0.967	3.369	KP
$L_{50}M_{50}$ 5 %	0.981	0.971	3.389	0.985	0.977	3.023	KP
$L_{50}M_{50}$ 10 %	0.951	0.926	5.141	0.966	0.949	4.260	KP
$L_{50}M_{50}$ 15 %	0.918	0.877	6.350	0.968	0.952	3.975	KP
C_{100} 5 %	0.926	0.889	7.520	0.930	0.895	7.332	KP
C_{100} 10 %	0.956	0.935	5.295	0.962	0.943	4.937	KP
C_{100} 15 %	0.935	0.903	6.180	0.953	0.930	5.240	KP
$C_{75}M_{25}$ 5 %	0.795	0.693	8.776	0.651	0.477	11.451	First-order
$C_{75}M_{25}$ 10 %	0.970	0.955	5.530	0.946	0.919	7.418	First-order
$C_{75}M_{25}$ 15 %	0.957	0.936	5.136	0.965	0.947	4.655	KP
$C_{50}M_{50}$ 5 %	0.967	0.950	6.054	0.954	0.931	7.126	First-order
$C_{50}M_{50}$ 10 %	0.950	0.925	7.698	0.914	0.871	10.074	First-order
$C_{50}M_{50}$ 15 %	0.979	0.968	4.220	0.977	0.965	4.396	First-order
Uncoated DAP	0.890	0.835	9.459	0.906	0.859	8.759	KP

highly crosslinked networks in linseed-rich coatings, as evidenced by FTIR analysis, which promote heterogeneous, semi-permeable polymer matrices where diffusion occurs concurrently with polymer relaxation (7, 20–22). Conversely, the improved fit of the first-order model in CM-coated DAP aligns with reports that castor-oil-derived coatings form thicker, more flexible films that regulate nutrient release primarily through diffusion processes (9, 23). Collectively, the variation in model fitting across formulations highlights the dominant role of coating, polymerization behaviour and film integrity in governing phosphorus release mechanisms, emphasizing coating properties as the primary determinants of nutrient-release kinetics (3, 24).

Conclusion

This study demonstrated that blending linseed, castor and mustard oils produces effective biodegradable coatings capable of significantly modifying the curing behaviour, structural properties and release characteristics of phosphorus fertilizers. Among all formulations, the 50:50 LM and 50:50 CM blends formed the most stable films, providing uniform coverage and sustained phosphorus release. FTIR and SEM analyses confirmed successful film formation without altering the fertilizer core, while kinetic modelling revealed that LM coatings predominantly followed anomalous (KP) release and CM coatings aligned with first-order dissolution. Overall, these oil-based coatings offer an environmentally compatible approach for developing controlled release phosphorus fertilizers with improved nutrient use efficiency.

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Authors' contributions

PKB conducted lab study and wrote original draft, DRB and VKS guided in conceptualization and writing, BBB and RB helped in supervision, editing and visualization, SD and AD participated in the design of the study and performed the statistical analysis, AB participated in coordination, TR, AS, AJ and AN helped in setting up the experiment and laboratory analysis. All authors read and approved the final manuscript.

Compliance with ethical standards

Conflict of interest: Authors do not have any conflict of interests to declare.

Ethical issues: None

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