Top Interview

Insight Into Requirements, Challenges and Innovations in Formulation and Application Technology

Nouryon
Formulation of Agrochemicals in High Electrolyte Systems
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Stepan: Next Generation Compatibility Agent for Today's Complex Tank Mix Systems

Over the past couple of decades, it has become increasingly common, if not necessary, to combine multiple crop protection products into one spray application. Beyond the obvious advantages of time savings and reduced application costs, this strategy combats the ever-present challenge of pest and pathogen resistance by employing multiple modes of action (MOA). Leading crop protection companies implement MOA by combining several active ingredients into one formulation. In cases of single-active ingredient formulations, growers will tank mix multiple crop protection products to achieve the same desired MOA effect.

Regardless of approach, today, spray tanks increasingly contain more active ingredients than in years past, leading to a delicate system between tank partners. Further complicating matters, growers will often use liquid fertilizer as a carrier in place of water to gain even more efficiency. Given the complexity of modern crop protection strategies, it’s not surprising to see a rise in compatibility issues surrounding tank mixtures.

These incompatibilities can occur between active ingredients, but more commonly arise when formulations are diluted into liquid fertilizer, where hydrophobic organic molecules and water-soluble salts are at odds with each other. These incompatibilities can present in many forms, including the formation of particles or gels that rapidly settle out of suspension, separation of the mixture into layers, or excessive foaming. As one might imagine, spraying an incompatible mixture can result in a loss of efficacy due to uneven distribution, poor performance, or crop injury. In severe cases, these issues can clog sprayers that lead to equipment shutdown and cleanout.

In the crop protection industry, compatibility agents are used to combat stability issues before and/or after they arise. Our research has determined that more than 75% of commercial compatibility agents use phosphate esters, a majority of which are based on nonylphenol ethoxylates (NPEs). NPEs have been the backbone of compatibility agents for decades and remain dominant in the market due to availability and low cost. However, while NPE-based compatibility agents are effective in simple tank mixtures, they have marginal performance in complex systems. As incompatible

Figure 1. Reaction schemes of phosphate esters.
systems have become more challenging, innovation has stalled in the development of new compatibility agents that can meet the demands of tank mix systems today. Furthermore, NPEs have well-known environmental and health concerns, resulting in their use being slowly phased out across the agrochemical industry.

Considering today’s complex nature of tank mixtures and regulatory concerns surrounding NPEs, Stepan saw a need to develop a next generation phosphate ester compatibility agent. We believe the market has yet to take full advantage of the immense potential phosphate esters offer, given the ability to tune monoester/diester ratios, select from a wide range of hydrophobes, and vary the degrees of ethoxylation (Fig. 1). Thus we launched an initiative to study these parameters in detail to find the optimal phosphate ester, then leveraged our experience in formulation development to further enhance performance with additional additives to produce a top market compatibility agent.

With access to a wide array of hydrophobes, we synthesized and screened more than 70 unique molecules, identifying trends for each parameter. Each iteration was evaluated in nine incompatible systems containing a formulated pesticide and fertilizer. Our screen included a variety of formulation types, including emulsifiable concentrates, suspension concentrates, and soluble liquids. Performance was assessed based on the number of incompatible systems a phosphate ester was able to improve.

The first parameter that we studied in detail was the phosphate ester’s monoester/diester ratio. Phosphate esters with either high monoester or high diester content were evaluated across multiple carbon chain lengths and moles of ethylene oxide. The results demonstrated that high monoester containing phosphate esters were equivalent or better than their high diester counterparts across all systems, regardless of hydrophobe or moles of ethylene oxide (Fig. 2). This suggests that the more hydrophilic nature of monoesters enhances solubility in high electrolyte environments. For the rest of the study, we chose to work with only high monoester phosphate esters.

Next, we studied the effect of ethoxylation across various carbon chain lengths. Performance was found to improve when a lower degree of ethoxylation was employed and was not dependent on carbon chain length (Fig. 3). As moles of ethylene oxide were increased, the effect on compatibility became more variable based on a specific incompatible system and phosphate ester molecule. There appears to be a minimum degree of ethoxylation required for water solubility and broad-spectrum performance, beyond which performance begins to deteriorate as ethoxylation levels become too high.

Figure 2. Comparison of monoester and diester counterparts at CN.

Figure 3. Effect of moles of ethoxylation across four carbon chain lengths (CM, CN, CO, and Cp).

Figure 4. Effect of carbon chain length across three levels of ethoxylation (EOX, EOY, and EOZ)
Last, we evaluated a range of carbon chain lengths at multiple degrees of ethoxylation. An optimal carbon chain length was identified that displayed peak performance for all ethoxylation levels (Fig. 4). Combined, these trends led to a final phosphate ester with high monoester content, a low degree of ethoxylation, and an optimized carbon chain length.

With our top performing phosphate ester defined, we leveraged our formulation expertise and extensive portfolio of inerts to further enhance the compatibility performance. The phosphate ester was paired with an array of secondary components, including emulsifiers, wetting agents, and other compatibility aids. Light scattering measurements were implemented to track instability over time and identify the most stable formulation candidate (Fig. 5). Ultimately, optimized secondary components were identified and found to significantly increase the performance of the phosphate ester, leading to our new tank mix compatibility blend, TOXIMUL® CT.

TOXIMUL CT was tested against commercially available compatibility products (Fig. 6a). The commercial products all contained phosphate esters as their primary component, many of whom relied on NPE-based formulations. The results demonstrate that TOXIMUL CT provided superior compatibility performance over current commercial solutions in high electrolyte systems. For situations where incompatibilities between active ingredients were observed, we also saw an improvement using TOXIMUL CT. For example, compatibility is significantly improved in an atrazine/paraquat system with TOXIMUL CT over competitive products (Fig. 6b).

In conclusion, TOXIMUL CT is an excellent option for solving difficult compatibility issues over a wide variety of tank mix systems. In the design of this new blend, we evaluated more than 70 unique phosphate esters and screened more than 2,000 incompatible systems to find the best product. TOXIMUL CT is an innovative solution that can help meet the demands of today’s increasingly complex spray tank mixtures.

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References: