Comparison of methods for determination of stability of emulsions

Prisco, R.C.B; Piedade M. F; Savoy, V.L.T
Laboratório de Química Ambiental, Instituto Biológico, Brasil
rprisco@biologico.sp.gov.br

INTRODUCTION - The stability test is performed on the formulations of emulsions and emulsifiable concentrate type, in order to ensure, with safety, the efficiency of the formulation.
An emulsion is a fine distribution of two immiscible liquids or partially soluble liquids with each other that has an external phase (solvent) and an internal phase (solute), and, most often a component of the emulsion is water. Therefore, emulsifiable concentrate is a solution that form emulsion when mixed with water and the mixture formed must remain as a stable emulsion for a certain period to ensure uniformity of application. This study compared two methods described in different versions of ABNT NBR 13452 products pesticides formulated as emulsifiable concentrate, in order to verify the similarity of the methodologies and generation of waste of the analytical procedure.
MATERIAL AND METHODS - We evaluated four samples of pesticides, two with density greater than 1g/cm3 and two with density lower than 1g/cm3, with seven replicates for each method, a total of 56 trials. For the methodology currently in force should be weighed directly into a 250mL graduated cylinder containing 200 mL water standard thermostated at 30℃ (± 1℃)and total hardness of 20mg/kg expressed as CaCO3, equivalent to 2.5 mL of the pesticide, taking into consideration the density. After weighing, complete immediately with water standard until 250 mL, cap the test tube and rotate it 180 degrees, reversing the beaker every 2 seconds, 30 times, and return it to its original position. For products with lower density than 1g/cm³, immediately transfer the contents of the beaker into a 100 mL conical tube with standard grounded stopper (without any holes in the upper body of it), according to ASTM D-1837, fill the tube until to the neck and, for products with density equal to or greater than 1g/cm³, immediately transfer the contents of the beaker into a conical tube until the volume of 100 mL. Leave the conical tube to stand in the thermostat at a temperature of 30℃(± 1℃), without vibration, and place it upside down when the density is less than 1g/cm3. Make reading after 1 hour. For the second method, the previous version of ABNT NBR 13452, add to the conical tube, which contains already 95mL of water standard with the same conditions as in the previous test, with volumetric pipette 1 ml of the pesticide, complete immediately with water until 100 mL, cover and rotate 180 degrees, inverting the tube every 2 s, 30 times. Return it to its original position and leave the conical tube to stand in the thermostat at temperature 30ºC (±1℃) without vibration. Make reading after 1 hour. For products with lower density than 1g/cm³ the volume of cream separated occurs at the top of the tube. In this case, fill with water standard the total volume of tube, put it upside down to stand in the thermostat and read after 1 hour. Reporting the results of both methodologies for testing the stability of the emulsion must be mentioned the occurrence of clear and specific separation of oil phase and/or aqueous and calculated the percentage of cream formation, considering the density of the product and the parameters are set the maximum limits of 1% v/v cream and the absence of clear and specific separation of oil phase and / or in aqueous

solution.
RESULTS - The results of this study showed that no phase separation occured in both formulations nor with higher density than 1g/cm³ nor with lower density . Formation occurred in cream products evaluated in both methodologies, but did not exceed the maximum recommended in the standard. The methods applied to products with lower density than 1g/cm³ had similar, whereas for products with higher density than 1g/cm³ there was a numerical agreement thus preventing the determination of statistical calculations and therefore the methodologies were not considered equivalent. The method described in ABNT NBR 13452, currently in effect, generate higher volume of residue analytical as it is syrup prepared is 250mL (sample + water), whereas the previous version of the spray volume is 100 mL (sample + water).
CONCLUSION - We conclude that it is important that further changes should be applied to the methodologies, the determination of the mass corresponding to the volume of which is added to 1 ml conical tube in the second method, so that further studies and the data generated can support the review the standard currently in force.