



Formulation and processing of recycled-low-density-polyethylene-modified bitumen emulsions for reduced-temperature asphalt technologies



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HIGHLIGHTS

- Waste-polymer-modified bitumen emulsions for sustainable construction technologies.
- Recycled-LDPE-modified bitumen emulsions prepared by an inline emulsification.
- Emulsion droplet size distribution and rheology affected by binder formulation.
- Emulsion residue with enhanced modification compared to its counterpart binder.
- Emulsification yields binder residues with a highly dispersed polymer phase.

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ABSTRACT

Sustainable asphalt technologies involving materials recycling and energy-saving approaches are growing demands. Among waste polymers, recycled low density polyethylene (LDPE_R) is a promising bitumen modifier. Technological and viscous flow tests showed enhanced performance in LDPE_R-modified bitumens (PMBs) above 4 wt% polymer. However, 4 wt% LDPE_R underwent phase separation within 1 h when PMB was stored at high temperature, affecting product industrial implementation. Aiming at preventing polymer separation and promoting energy-saving technologies, bituminous binders (containing 2–5 wt% LDPE_R) were successfully dispersed as oil-in-water emulsions (O/W) by an inline emulsification procedure at controlled temperature and pressure. Emulsions with up to 63 wt% bitumen phase exhibited broad droplet size distributions and non-Newtonian flow behaviors strongly affected by the emulsion and PMB formulations. Optical and calorimetric techniques conducted on emulsion bituminous residues showed that shear conditions during emulsification increased dispersion of the swollen polymer phase, which led to better properties than the parent PMBs at high in-service temperature.

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1. Introduction

The traditional asphalt mixtures (composed by mineral aggregates and bitumen) are always produced at high temperatures, usually above 150 °C, so that the low binder viscosity assures the adequate coating of the aggregates, giving rise to the so-called hot-mix asphalt technology (HMA). Alternatively, different types of reduced-temperature technologies have emerged over the last decades, which involve less energy consumption and lower fume and particle emissions to the environment (Lesueur, 2011). In

particular, cold-mix asphalts (CMA) consist in coating the mineral aggregates with an emulsion formed by very fine bitumen droplets dispersed in water, and stabilized by a surfactant at a concentration of about 1 wt% (Gringras et al., 2005). Consequently, bituminous emulsions encompass a reduction in the temperature requirements with respect to traditional hot asphalt mixtures, while increasing flexibility by extending working time for transport and application, and reducing ageing of the binder during the production (Carrera et al., 2015).

Unfortunately, even the best design and constructed road pavements deteriorate due to the combined effects of traffic loading and weathering. The most common distresses at high in-service temperatures are rutting (permanent deformation under heavy vehicle loading) and fatigue cracking (cracks due to repetitive

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application of loads). Furthermore, thermal cracking, related to cracks due to the lack of asphalt flexibility at low in-service temperatures, is also a common problem of road pavements (Cuadri et al., 2013, 2014a; Lu et al., 2003; Read and Whiteoak, 2003). To prevent such issues, bitumen performance has been traditionally improved through the addition of virgin polymers such as SBS, SBR, LDPE, EVA, etc. Alternatively, waste polymers (from plastic from agriculture, crumb tyre rubber, etc.) are considered promising modifiers that may provide further economical and environmental advantages (Cong et al., 2015; Ho et al., 2006; Soudani et al., 2016; Yan et al., 2015; Yousefi, 2003). However, the bitumen modification by high molecular weight polymers may yield a significant phase separation during their storage at high temperatures (Garcia-Morales et al., 2004a).

On these grounds, a way of improving the performance of standard cold-mix asphalts, preventing polymer phase separation drawbacks, is through the use of modified bituminous emulsions, in which polymer modification may take place either before or after binder emulsification (Lesueur, 2011). Thus, polymer modified bitumen emulsions (PMBEs) can be found as either dispersions of polymer modified bitumen (PMB) or as latex modified emulsions. In the first category, the polymer is pre-blended into bitumen before emulsification process. In the second one, latex (stable dispersion of polymer microparticles in aqueous medium) is added to a neat bitumen emulsion, either prior to the colloid mill or after (latex pre- or post-addition method), so that a residue of PMB is obtained after emulsion drying (Lesueur, 2011).

In this context, present work is part of a research project with the overall objective of promoting more sustainable construction technologies, throughout the use of recycled polymers (e.g. urban wastes such as LDPE) and the application of energy-saving technologies such as cold mix asphalt (CMA), based on bituminous emulsions. However, the practical use of recycled LDPE as bitumen modifier is hindered by the poor storage stability of the resultant PMBs, due to a quick polymer separation when stored at high temperature (Singh et al., 2013).

With the aim of overcoming that serious drawback (alongside the development of energy-saving materials), this work assesses the feasibility of obtaining stable emulsions from PMBs prepared with recycled low density polyethylene (LDPE_R), by using an inline emulsification procedure. It is worth noting the processing and formulation of such PMBEs has not been reported before. Furthermore, this study addresses the effect that variables such as polymer concentration in the PMB and PMB concentration in the emulsion (i.e. disperse phase content) exert on emulsion properties and on PMBs obtained before emulsification and after emulsion drying. To that end, steady viscous flow curves, droplet size distribution, Differential Scanning Calorimetry (DSC) tests and optical microscopy analysis were conducted on these samples.

2. Experimental

2.1. Materials

Bitumens, supplied by Repsol S.A. (Spain), with penetration grade 160/220 and 70/100 (referred to as B160/220 and B70/100, respectively) were used as base materials for polymer-modified bitumen (PMB) and emulsion preparations. Ring and Ball Softening Temperature ($T_{R&B}$) and penetration values (measured according to EN 1426:2007 and EN 1427:2007, respectively) are gathered in Table 1 for both bitumens.

Recycled low density polyethylene (LDPE_R) from industrial origin, kindly donated by Cordoplas S.A. (Spain), was selected as bitumen modifier. As may be seen below, LDPE_R is blend of two immiscible polymers: low density polyethylene (LDPE) and linear low density polyethylene (LLDPE). The degree of crystallinity, calculated from its heat of crystallization and the theoretical value for a fully crystalline material, was found to be 28%. Furthermore, a styrene-butadiene-styrene copolymer (SBS) Calprene[®] 501 (Dy-nasol) was also used as reference bitumen modifier.

Finally, an alkyltrimethylenediamine derived from N-tallow, with average molecular weight of 340 g mol⁻¹ and melting point between 30 and 40 °C (provided by Kao Chemicals, Spain), was used as emulsifier for the preparation of cationic bituminous emulsions with quick and medium breaking rate in contact with the aggregate.

2.2. Preparation of the polymer modified bitumen emulsions (PMBEs)

The preparation of the PMBEs consisted of two steps, an initial bitumen modification with recycled LDPE to obtain the PMB and its subsequent oil-in-water emulsification to give the PMBE.

2.2.1. Formulation and processing of the bituminous and aqueous phases

Bitumen modification by LDPE_R was conducted with a Silverson (U.K.) high-shear homogenizer, under optimized blending conditions of 170 °C, 1 h and 5000 rpm. In previous experiences, different LDPE_R bituminous samples were prepared varying the temperature/time/rotational speed conditions. Among them, optical microscopy observations conducted on them demonstrated that 170 °C/1 h/5000 rpm values results in binders with a better polymer distribution. Blends of both bitumens (B160/220 and B70/100) and recycled LDPE at the concentrations ranging from 2 to 5 wt% polymer were prepared. So, for illustrative purposes, a bitumen B160/220 modified by 4 wt% LDPE_R will be referred to as "B160/220PMB-4", hereinafter. In addition, a bituminous reference sample with 3 wt% SBS (formulation typically used in paving applications) was prepared at 180 °C for 1.5 h and 5000 rpm (Airey, 2003), with the same homogenizer.

Emulsion aqueous phase was prepared by dispersing 2.5 wt% emulsifier into distilled water at pH 1 in order to polarize

Table 1
Ring and ball softening temperature ($T_{R&B}$) and penetration of LDPE_R-modified bitumens.

	$T_{R&B}$ (°C) ^a LDPE _R (wt%)					Penetration (1/10 mm) ^a LDPE _R (wt%)				
	0	2	3	4	5	0	3	4	5	
B70/100	45 ± 0.6	–	50 ± 1.2	61 ± 1.4	> 74	73 ± 3	63 ± 2	50 ± 2	41 ± 2	
B160/220	40 ± 0.5	44 ± 0.6	–	66 ± 1.4	68 ± 1.5	179 ± 5	–	94 ± 3	74 ± 2	

^a Values given as: mean ± standard deviation.

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