A compilation of laboratory methods for studying stability of bitumen emulsions

U. Isacsson, Ph. D.,
Swedish Road and Traffic Research Institute, Linköping.

A compilation of methods for characterizing the stability of bitumen emulsions is presented. The compilation covers both methods for measuring stability during transport, storage and handling of an emulsion, in addition to methods for measuring breaking and hardening in various applications. A general description of each test method is given.

The concepts of "breaking" and "curing" are defined. Factors which may affect the stability of an emulsion are described.

General views on the value of certain test methods and the orientation of further development work considered necessary in this area are expressed.

1. INTRODUCTION

Stability is a basic property of bitumen emulsions used for road building. From a general point of view, stability should be as high as possible during storage and transport. On the other hand, the bitumen should be deposited on the aggregate as soon as possible after spreading on the road. These two demands counteract each other. Therefore a compromise must be sought in the formulation of an emulsion. To achieve this, it is necessary to have access to suitable laboratory methods for studying the stability of the emulsion.

Since the work by Weber and Bechler [1] was published half a century ago, a great number of methods for characterizing the stability of bitumen emulsions have been proposed. The purpose of this paper is to provide a compilation of such methods. Even if the list of methods is long, it is probably not complete. There are very probably other methods not mentioned here which are used, for example, by manufacturers of emulsifiers.

2. BASIC CONCEPTS

In discussions between road engineers concerning the stability of bitumen emulsions, one or more of the concepts of breaking, setting and curing are normally used. Unambiguous definitions of these concepts have not been found in the literature. The following proposals constitute an attempt to remedy this shortcoming.

It is stated first that the three concepts mentioned above are used rather interchangeably in the literature. A case in point is the Asphalt Institute Manual for Asphalt Emulsions [2] where breaking and setting are regarded as synonymous concepts. In this paper only the terms breaking and curing will be discussed. It should also be noted that breaking (on the basis of the definition given below) is a change in an emulsion while the curing process is defined on the basis of a binder-aggregate system.

In order to illuminate the following definition of breaking, it may be appropriate first to define a number of fundamental concepts (see fig. 1).

A bitumen emulsion is a dispersion of bitumen in water. The process causing the bitumen droplets to form an agglomerate is called coagulation or flocculation. These terms are often used interchangeably, but it is useful to introduce a distinction between coagulation, implying the formation of compact aggregates leading to the macroscopic separation of a coagulum, and flocculation, implying the formation of a loose or open network which may or may not separate macroscopically [3]. A coagulated emulsion is therefore more difficult to redisperse than a flocculated emulsion. The process causing the bitumen droplets to merge into larger droplets is termed coalescence.

In practice it may be difficult to distinguish between flocculation, coagulation and coalescence. According to the opinion of the RILEM Committee 56 MHM "Hydrocarbon Materials" it is sufficient to use flocculation and coalescence as defined above when discussing the stability behaviour of bitumen emulsions. Consequently, coagulation can be omitted without disadvantage.

In this document, breaking of an emulsion is defined as a process in which coalescence of bitumen droplets takes place. The breaking process begins when the first droplets...
merge into larger droplets and ends when the bitumen has become a continuous phase, which may contain enclosed water droplets (cf. fig. 1).

When discussing the breaking of an emulsion, the concepts of sedimentation and creaming (see fig. 1) are very often mentioned. Sedimentation or creaming may occur if the bitumen density differs from the water phase density. A sedimented (or creamed) bitumen emulsion may (but not necessarily) have flocculated and is therefore in principle redispersible, for example through stirring. In practice, however, the occurrence of sediment during storage, for example, is always undesirable since the result of redispersal depends to a large extent on how this has been carried out. If strong shear forces are used, the energy applied may cause the bitumen droplets to coalesce.

The definition of the concept of curing presupposes that the emulsion has come into contact with mineral aggregate (see fig. 2). The curing process which implies the removal of water (and possibly solvent) starts, by way of definition, when the emulsion and mineral aggregate have come together and ends when all the water (and solvent) has been removed. The water leaves the system in liquid and gaseous form.

In figure 2, three stages in the overall process are schematically illustrated. In the first stage, emulsion has just been mixed with aggregate and this is by definition the beginning of the curing process. The breaking process may have started before this stage as breaking may occur in the emulsion itself. In stage 2, the breaking process is ended as a continuous bitumen phase has been formed. Finally, in stage 3, where all the water (and solvent) has left, the binder-aggregate is fully cured, and, consequently, the overall process ended.

From the definitions of breaking and curing given above, it follows that:
- a bitumen emulsion may break before it has been mixed with mineral aggregate;
- a partially broken emulsion, i.e. an emulsion which has begun to coalesce, may be satisfactory in practice, provided that the coalescence has not proceeded too far;
- the breaking and curing processes in general take place in parallel.

3. FACTORS WHICH MAY INFLUENCE THE STABILITY OF BITUMEN EMULSIONS

The concepts of breaking and curing defined in the preceding section are very complex phenomena. No attempt will be made here to summarise the mechanisms of the breaking and curing processes proposed over the years. However, a summary of factors which may influence these processes may be appropriate in order to illustrate their complexity.

Factors which are significant in maintaining the stability of an emulsion during transport, storage and handling

- design of the transport and storage tank;
- movement of the tank during transport;
- degree to which the tank is filled;
- temperature of the emulsion;
- method for maintaining the homogeneity of the emulsion (stirring, pumping);

Factors which are significant in maintaining the stability of bitumen emulsion during transport, storage and handling

- design of the homogenization equipment (e.g. the pump or stirrer);
- characteristics of the emulsion (cf. below).

Factors which affect breaking and curing in a given application

- The characteristics of the emulsion:
  BITUMEN, origin, hardness and content;
  EMULSIFIER, type and content;
  WATER, pH and electrolyte content;
  PARTICLE SIZE DISTRIBUTION OF THE EMULSION (which in turn depends on the above characteristics of the bitumen, emulsifier and water);
  TEMPERATURE OF THE EMULSION.
- The characteristics of the aggregate:
  TYPE (mineralogical composition);
  SURFACE, form, surface structure, porosity, particle size distribution, filler content and clay content;
  WATER CONTENT.
- Quantity relationship between emulsion and mineral aggregate.
- Climatic condition, temperature, humidity, wind speed.
- Factors in force at the time of application:
  METHOD OF SPREADING THE EMULSION in surface treatments;
  MIXING PROCEDURE AND MIXING TIME when manufacturing the emulsion concrete;
  COMPACTION WORK.

The above list of factors is undoubtedly incomplete, but should be sufficiently comprehensive to indicate the complexity of the subject. This is also illustrated by the large number of methods (over 50) proposed for determining the stability of bitumen emulsions (see next section).

4. LABORATORY METHODS

It is rather complicated to group the methods in a meaningful manner. Whatever the basis of the division, it may be difficult to place one or more methods in the correct position.

In this compilation, the methods are divided into two main groups, namely methods where aggregate is not used (table I) and methods with aggregate (table II). The first main group may in turn be subdivided into chemical