

Determination of LAS by HPLC method

LAS'ın HPLC metodu ile tayini

**Hüseyin Koç, Tuncay Gezgin
and
Kasım Cemal Güven***

¹University of Istanbul, Institute of Marine Sciences and Management
Vefa , Istanbul, Turkey,

Abstract

LAS was determined by HPLC method and the results were compared with those obtained by MBAS method. The standard curve was plotted from the total areas of the peaks of LAS. HPLC methods was precise and reliable in determination of LAS.

Keywords: LAS, HPLC, MBAS techniques.

Introduction

LAS is a basique compound of detergent. Many papers were published on its pollution, toxicitiy and determination in enviroment. LAS is a complex mixture of C₈-C₁₆ alkyl benzene sulfonate. It is prepared by sulfonation of LAB. The distribution of alkyl chain derivatives of LAB is 25 % phenyl C₁₀, 0.4% phenyl C₁₀, 8,9 % (max. 15), phenyl C₁₀ + phenyl C₁₁, 30-55% (max.) phenyl C₁₁, 33.7%

* Corresponding Address.

phenyl C₁₂, 31% phenyl C₁₃, 24% phenyl C₁₃ + phenyl C₁₄, 30% (max.) phenyl C₁₄ 2% (max. 6). Beside these elements it contains 2 phenyl alkanes, tetralins and indans. Total amounts of linear alkyl benzene and branched alkyl benzene were 95-99 and 3-4 % successively. The main problem was the sulfonation form benzene ring of LAB. During the process oxydation occurs, polimerization and decomposition of molecule take place. The little amount of LAB remains in unsulfonated form. After sulfonation various isomers are produced thus endincating that LAS contains many derivatives.

The determination of LAS was made by various methods such as : potentiometric (He *et al.*, 1993), spectrometry; methylene blue active substance (MBAS) (Standard methods), metachromatic (Güven *et al.*, 1994; Akıncı *et al.*, 1997), IR (Helmann 1978,), AAS (Crips *et al.*, 1976; Barrait *et al.*, 1984), GC/MS (Raymunda and Preston,1992; Eganhouse *et al.*, 1983; Hon . Hami *et al.*, 1978,1980), HPLC (Marcomimi and Grigen, 1987; Terzic and Ahel, 1993). HPLC technique is based on the measurement of the area of LAS peaks in chromatogram.

In this work the determination of LAS by HPLC method was an investigated and the results were compared with those obtained by MBAS method.

Material

LAS (95.6%) was obtained from LEVER, İzmit, Turkey.

Stock solution : 0.100 g LAS was dissolved in 100 ml distilled water

LAS solution: 0.1, 0.25, 0.50 and 1 ml stock LAS solution were diluted to 1/100

Apparatus: HPLC (HP 1100)

Method

1. Calibration curve

The calibration curve of LAS was plotted as:

1.1. by MBAS according to Standard method: The concentrations use were 10-100 ug/L

1.2. by HPLC method: 10, 25, 50 and 100 ug/L LAS solution were applied to HPLC apparatus.

Column : 100x2.1 mm Hypersil ODS 100-5um

Detector: Diode-Array. λ_{\max} 222 nm

Mobile Phase: A: 0.005 M KH₂PO₄

B: Acetonitrile

Flow rate : 0.5 ml/min.

Time(min)	A%	B%
0.00	74	26
25	0	100

The area of peak for each concentration was taken from apparatus. Than the calibration curve was plotted from the total area for each concentration.

Result

The standard equation of calibration curve of LAS through MBAS method is:

$$y = 0.0038 \times C + 0.0196 \quad r^2 = 0.996$$

The calibration curve of LAS through HPLC is shown in

Fig. 1. Its standard equation is :

$$y = 62.134 \times C + 15.554 \quad r^2 = 0.999$$

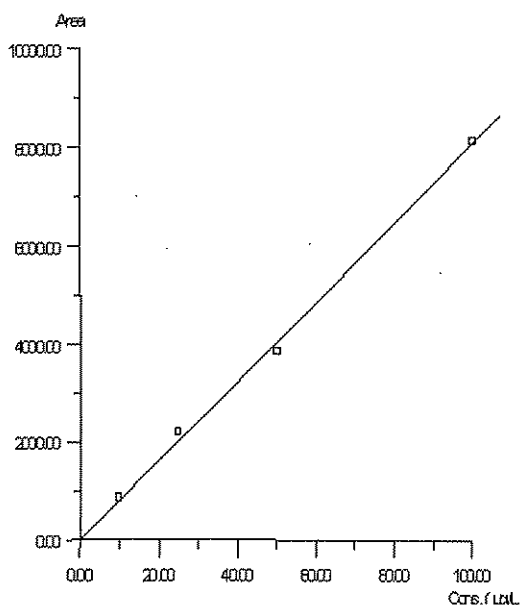


Fig.1: The standard curve of LAS by HPLC method.

HPLC chromatogram of LAS showed 12 peaks. HPLC chromatogram of LAS in a concentration 10, 25, 50 and 100 ug/L are shown in Fig. 2.1. and 2.2.

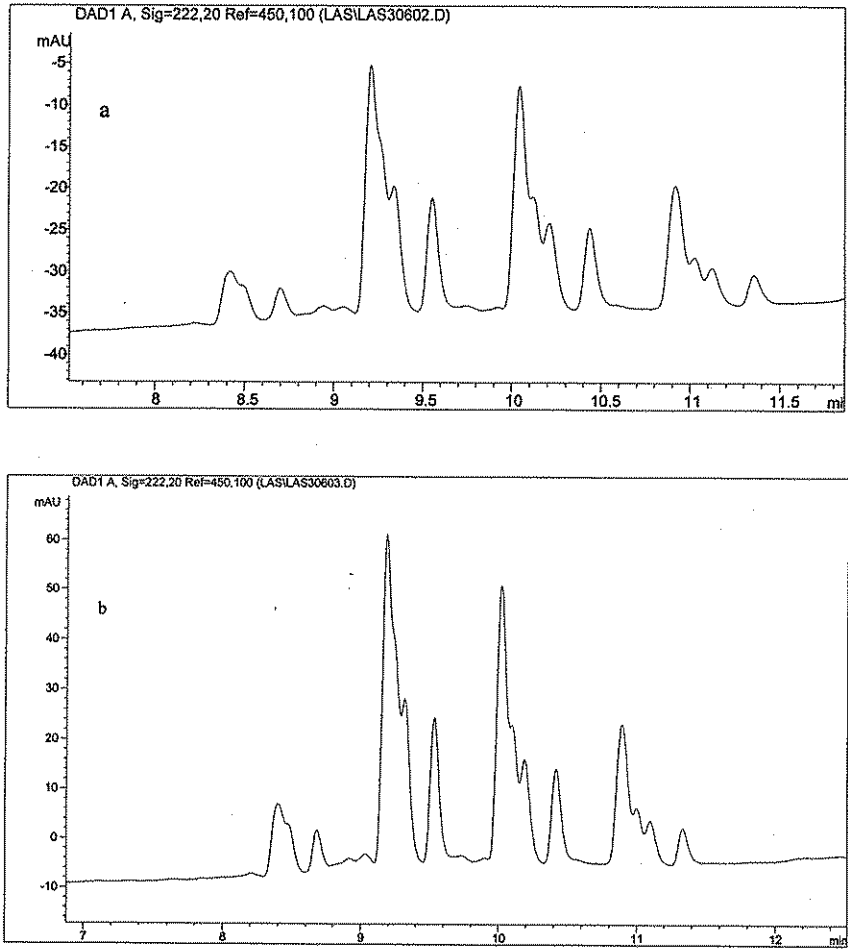


Fig. 2.1. HPLC Chromatogram of LAS in water ; a: 10µg/L, b: 25 µg/L

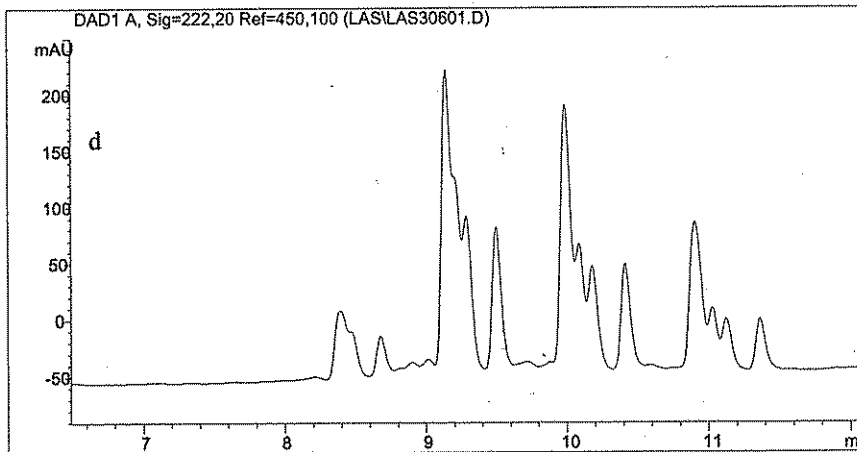
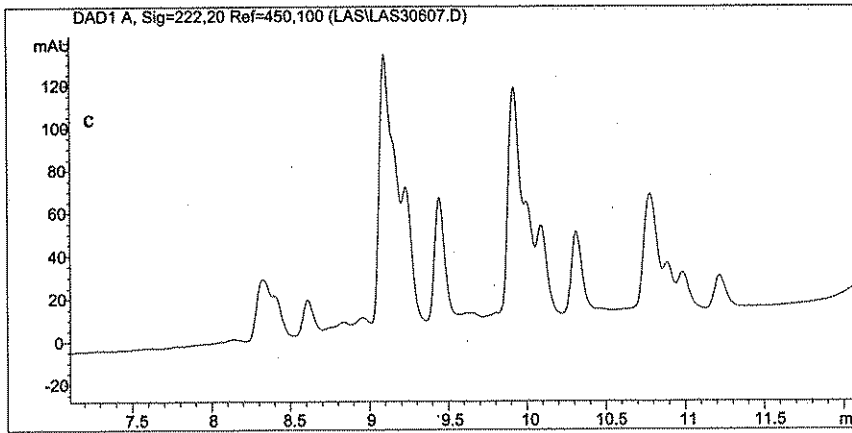


Fig.2.2. HPLC Chromatogram of LAS in water ; c: 50µg/L, d: 100 µg/L

The retention times of peaks are:

	Min
1	7.518
2	7.826
3	8.367
4	8.499
5	8.719
6	9.206
7	9.364
8	9.584
9	10.045
10	10.142
11	10.231
12	10.451

The results of LAS amount obtained by MBAS and HPLC methods are shown in Table 1.

MBAS	HPLC
10.68	12.45

Table 1. LAS amount found by MBAS and HPLC method (mg/L). LAS added : 12,5 mg/ L

As can be seen in the table the results of HPLC method gave best results. Each technique has some difficulties in the determination of LAS. However MBAS technique is commonly used. Some organic and inorganic compounds interfere with the results. Metachromatic method (Güven *et.al* 1994) has also the same problem. AAS and IR technique showed difficulties. GC/MS and HPLC methods were not easy. The problem of HPLC determination is the origin of LAS . It is not a homogenous material. It contains many alkyl chain benzene derivatives and its composition varied depending on the manufacturer of LAB and also the sulfonation process applied. The proposed HPLC method and MBAS technique gave not the same results. On the other hand HPLC chromatogram was not the same for all LAS products. The

results of MBAS method were not also similar in determination of LAS by various laboratories. The same problem was also observed in the analyses of duplicate samples.

LAS that contained less than 9 carbon and greater than 14 carbon atoms were negligible both in the aquatic environment. C₁₀-C₁₃ was regarded as the total amount of LAS. The major components of LAS were C₁₁-C₁₂. The advantage of HPLC technique is that all the components of LAS can also be determined.

HPLC technique can be used beyond to MBAS technique for the LAS determination in the environment studies.

Özet

Bu çalışmada deterjanın temel maddesi LAS'ın suda HPLC metodu ile tayinine ait bir teknik önerilmiştir. Bulgular MBAS metodu ile elde edilen sonuçlar ile karşılaştırılmıştır. LAS'ın HPLC de miktar tayini için kromatogramdaki 12 pikin toplam alanı kullanılmıştır. İncelenen HPLC ve MBAS metodları arasında sonuçları bakımından HPLC tekniğinin daha hassas olduğu saptanmıştır.

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