

Analysis of a Liquid Professional Cleaning Product, and Polycarboxylates Detected Among its Components

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Abstract

A Greek liquid cleaning product was analyzed and its components were identified with FT-IR spectroscopy. The soluble-insoluble matter ethanolic 95° of a professional cleaning product was determined quantitatively to be 3.15%w/w, and 1.21%w/w, respectively. The FT-IR spectra were recorded of both the insoluble matter ethanolic 95° and the soluble matter ethanolic 95°, of the liquid professional cleaner. The anionic-active matter content of the product was also determined quantitatively to be 0.52%w/w. Polycarboxylic acids salts, present in the detergent product, were identified qualitatively from the ir peaks at 2912cm⁻¹, at 1723cm⁻¹, at 1567cm⁻¹, at 1402cm⁻¹, at 1334cm⁻¹ and at 1128cm⁻¹ which acted as sequestering builders. The anionic surfactant present was identified primarily from the peaks at 1182cm⁻¹ and at 1049cm⁻¹. The non-ionic surfactant present was identified from the peaks at 1121cm⁻¹ and at 1011cm⁻¹. The fragrance molecule was identified qualitatively from the ir peaks at 934cm⁻¹, at 832cm⁻¹, at 774cm⁻¹, at 622cm⁻¹ and at 608cm⁻¹. An anion exchange resin column was used to elute the anionic surfactant with ammonium bicarbonate solution in an isopropanol/water mixture 60/40v/v. The non-ionic surfactant eluted at the end with isopropanol. Primary biodegradability, and then final aerobic biodegradability tests of the cleaner broke down the surfactants and finally decomposed them into carbon dioxide, water and microbial cellular components.

Keywords: Cleaning product; Polycarboxylate polymer; Anionic surfactant; Non-ionic surfactant; Fragrance; Detergent under basic conditions; Electrophiles; Nucleophiles; FT-IR spectra; Anion exchange resin column; Biodegradability

Introduction

A detergent contains one or more surfactants formulated with more than one component to enhance the process of cleaning, where removal of soils is difficult due to the strong attraction of soil with fabrics, and poor penetration and adsorption of surfactants onto the soil and fabric interface [1]. Sequestering builders, such as polyphosphates inactivate water hardness mineral ions and hold them tightly in solution [1]. There are also the precipitating builders, such as sodium carbonate which remove water hardness ions [1].

Polycarboxylate polymers are developed to replace the functions of phosphates in cleaning products and to enhance the efficiency of surfactants by preventing precipitation via interactions with hard water ions [2]. Polycarboxylates enhance the cleaning performance of a detergent product by many ways such as softening the water, preventing scale buildup, dispersing soils and stabilizing formulations by making them key multifunctional additives in the professional cleaning system [2]. Different molecular weight polycarboxylates can be used as an effective remedy for builder's ability improving in sodium-carbonate/ zeolite detergent formulations and therefore secondary washing performance of laundry detergents

[3]. Polycarboxylates show a marked superiority to STPP (sodium tripolyphosphate) in their ability to sequester calcium ions, prevent incrustation of fibers and re-dissolve calcium salt precipitates [3]. A sequenstrant builder is incorporated in an anionic detergent product formulation for a specific purpose of preventing the loss of anionic surfactant through precipitation as its insoluble calcium salt [3]. Also, except from laundry detergents, superplasticizers, like polycarboxylate ethers or polycondensates are used to control and adjust the rheological properties of cementitious materials [4]. A most advanced concrete superplasticizer technology is polycarboxylate, a comb-like copolymer, which adsorbs on particle surfaces via the polycarboxylate backbone and against cement grain agglomeration via the steric hindrance of polyethylene oxide side chain [5-6]. Fourier-Transformed Infrared (FTIR) spectroscopy and also dynamic light scattering are applied to investigate the molecular structure of polycarboxylate-based superplasticizers which are frequently used in concrete technology [7-8].

The cleaning performance of cleaners is based on surfactants. Besides anionic and non-ionic as well as cationic surfactants, organic acids, such as citric acid, and fragrance, the cleaner contains lower alcohols, such as ethanol or 2-propanol and water [9]. FT-IR spectroscopy allows the identification of compounds of the detergent formulation and delivers information about which types may be present and which can be excluded [9]. In addition to FT-IR spectroscopy LC (liquid chromatography) is the analytical tool for the theoretical and experimental investigation of detergent mixtures. More specifically in a reversed-phase HPLC [10] the non-ionic surfactants could elute later than the anionic surfactants, due to their higher hydrophobic interaction with the nonpolar stationary phase, such as C18 silica. The anionic surfactants with sulfate head groups elute earlier, since they exhibit stronger affinity for the aqueous mobile phase. Using calibration standards, validation of the retention times is achieved, and finally with detection using UV-Vis we could identify and measure the concentration of the components of the complex mixtures of liquid cleaners.

Also, appropriate measures regarding detergents should ensure a high level of environmental protection, especially regarding the aquatic environment [11]. The surfactants contained in a detergent mixture must comply with the biodegradability criteria as laid down in Regulation (EC) No 648:2004 [11] on detergents.

Materials and Methods

Instruments and materials

Materials

The liquid detergent tested in this study was a Greek product, named blue velvet Professional 1L and it was a general purpose super cleaner, odorless, with barcode: 5200115500029. Among others it contained less than 5% anionic surfactants, less than 5% non-ionic surfactants, sodium hydroxide and aromatics (fragrance molecules), as shown on the product label.

Ethanol absolute 2.5L UN 1170, was from Carlo Erba, Hyamine 1622 solution, 0.004mol/l was from Merck Germany, HCl 0.1N, UN

1789, Product code 4042, was from Carlo Erba, Phenolphthalein 50g, Chim. Pure, was from Riedel-De Haen AG, Made in Germany, NaOH 0.1N was from Carlo Erba, RPE, UN 1824, CHCl₃ 1L was from PENTA, stabilized with ~1% ethanol A.G. Mr. 119.38, barcode:8595142231112, 2-propanol 2.5L Fisher Scientific UK Batch 0879418, Code:P/7500/17, Methanol ≥99.8% HPLC grade, Lot:2051871, 2.5L Fisher Chemical, solution of NH₄HCO₃ 60/40v/v (0.3 mol NH₄HCO₃ in 1000ml mixture isopropanol/water 60/40v/v), anion exchanger Dowex 1x2, 500g Serva 41011 (50-100mesh) counter ion Cl⁻, Lot 15113, D-69115 Heidelberg Feinbiochemica GmbH & Co, HCl 10%w/w. Ethyl alcohol and the other chemicals were used without further purification. For all the experiments preparations deionized water was used.

Apparatus

A pH-meter Metrohm 716 DMS Titrino, Swiss made was used for measuring the pH-value of the detergent product. The analytical balance Mettler Toledo AB 204-S/FACT accurate to 0.1mg, with maximum capacity 220g was used to weigh the detergent sample amounts for the analyses. The oven Memmert direkt, capable of being controlled at 103 °C ± 2 °C, was used to dry the sample until constant mass. The water bath FALC, 220/240V, 50Hz was used to heat the sample solutions. The FT-IR spectrometer Perkin Elmer Inc Spectrum 2000 Version 5.0.2 Copyright 2004, was used to record the FT-IR spectra. Ordinary laboratory apparatus was used like beakers of capacity 250ml and of capacity 3000ml, one-mark volumetric flasks of capacity 500ml, conical flasks of capacity 250ml, a burette of capacity 50ml. Also, exchanger columns with heating jacket and cock, inner tube diameter 60mm and height 450mm, rotary evaporator.

Analysis of the detergent product

Determination of soluble -insoluble matter ethanolic 95° of the detergent sample blue velvet Professional 1L

We weighed 15.1656g of the detergent sample blue velvet 1L, in a 250ml beaker and we added to it 75ml ethanol 95%w/v and then we heated on a water bath for 2 hours. We dried a filter paper, in parallel, to be used for the filtration of the insoluble matter content, in the oven controlled at 103° ± 2 °C, for 1hour. We allowed it to cool in a desiccator and we weighed it to be 1.0362g. We placed it in a funnel mounted on a glass container on the water bath. We decanted the supernatant liquid of the detergent on to the filter paper. After decantation we added 25ml of ethanol 95%w/v to the 250ml glass beaker and after heating it to near its boiling point, we transferred the insoluble matter to the filter paper. We dried the filter paper and the residue on it, in air and we placed it in the oven at 103° ± 2 °C. After 1hour we removed the filter paper, we left it in the desiccator for it to cool and we weighed it. The yield was 0.1834g, or 1.21%w/w of insoluble matter ethanolic 95° of the detergent sample blue velvet professional. The ethanolic solution of the filtrate in the glass container on the water bath was then heated until it fully evaporated and the glass container with the soluble matter was heated to constant mass in the oven at 103° ± 2 °C. We cooled it in a desiccator and the contents were weighed. The yield

was 0.4770g, i.e. 3.15%w/w of soluble matter ethanolic 95° of the detergent sample blue velvet Professional 1L.

Determination of the anionic-active matter content of the detergent sample blue velvet professional 1L, by manual direct two-phase titration procedure [12]

According to the International Standard ISO 2271:1989 [12], we weighed 16.8687g of the detergent product blue velvet professional 1L, into a 250ml beaker, which was a sample amount containing 0.004mol of the anionic-active matter content. Then, we transferred it to a 500ml one-mark volumetric flask with ground glass stopper and we diluted to the mark with deionized water. We mixed it up thoroughly and we transferred 20ml of this solution to a measuring cylinder by a pipette. The endpoint of the ensuing titration, with hyamine 1622 0.004M, according to ISO 2271 [12], was achieved at the moment when the pink colour was completely discharged from the chloroform layer, which became a greyish blue color [12]. The volume of benzethonium chloride solution used was 2.5ml. The %w/w of the anionic active matter content was calculated as follows: $\frac{2.5\text{ml} \times 0.004\text{M} \times 348.49\text{g/mol} \times 5}{33.7374\text{g}} = 0.52\%w/w$, where 348.49g/mol, was the approximate molecular weight of the anionic surfactant.

The yield was 0.52%w/w $\pm$ 0.02%w/w of anionic active matter content, from 33.7374g of the detergent sample blue velvet professional 1L, in 1Lt water deionized solution.

Determination of base

We weighed 1.0088g of the detergent product blue velvet professional 1L, into a 250ml conical flask and added 10ml of deionized water. We titrated with the burette of 50ml capacity, and with 0.1N HCl. Phenolphthalein was used as an indicator. It was determined that the base content was 0.32%w/w, expressed as NaOH, in the detergent product blue velvet professional 1L.

Anion exchange resin column-procedure [11]

First 600ml of anion exchange resin were placed in a 3000ml beaker and finally covered with the addition of 2000ml of deionized water. The resin was allowed to swell for two hours and was transferred to the column using deionized water. The column was capped with glass wool. The column was washed with 5000ml solution of 0.3mol ammonium bicarbonate in mixture isopropanol/water 60/40v/v, until the chlorine was gone. Then the column was washed with 2000ml of deionized water. Then the water was displaced with a 2000ml isopropanol/water mixture 60/40v/v, with a flow rate of 30ml/min. The exchange column was in the OH form and was ready for use. The exchange column was heated to a temperature of 50 °C with a thermostat. Then the soluble matter ethanolic 95°, 0.4770g was extracted with an isopropanol/ water mixture 60/40v/v. This solution was heated to 60 °C and passed through the exchanger resin at a flow rate of 20ml/min. Then the column was washed with 1000ml of warm isopropanol/water mixture 60/ 40v/v. The anionic surfactant was eluted from the anion exchanger column with 5000ml of the ammonium bicarbonate solution. The eluate was evaporated to dryness on the steam bath.

The non-ionic surfactant remained in the column and was eluted at the end with pure isopropanol and was evaporated to dryness.

Biodegradability of the surfactants of the cleaning product mixture [11]

The performance of the biodegradability tests required the separation and isolation of the anionic surfactant from the non-ionic and the soap. The fatty acids of the soap were separated by extraction with ethanol containing CO₂. Then, the anionic surfactant, obtained above as ammonium salt, by elution with ammonium bicarbonate solution in the isopropanol/water mixture, was used for the biodegradability testing. First, the primary biodegradability was controlled which was the change of the structural type of the surfactant by microorganisms, which resulted in the loss of its surfactant property due to the breakdown of the parent substance. In the biodegradability laboratory then, took place the final aerobic biodegradation where the surfactant was broken down by microorganisms in the presence of oxygen. The surfactant was decomposed into carbon dioxide, water and inorganic salts of any other element present, and in new microbial cellular components (biomass).

Results and Discussion

We measured the pH value of the detergent solution blue velvet professional 1L, in the Metrohm 716 DMS Titrino pH-meter, as it is, to be pH 11.73 $\pm$ 0.3 at 22.1 °C [13]. The infrared spectra of soluble and insoluble matter ethanolic 95°, of the detergent sample were recorded in the solid-state using diamond ATR. Firstly, we recorded the FTIR spectra of the insoluble matter content ethanolic 95° (Figure 1), of the detergent product blue velvet professional 1L.

In Figure 1 the FT-IR spectrum of the insoluble components in ethanol 95° of the liquid cleaner was given, which was determined to be 1.21%w/w of the mixture of the detergent product blue velvet 1L. In the FT-IR spectrum in Figure 1, polycarboxylic acid salts were identified to be present, in the detergent product blue velvet 1L, from the peaks at 2912cm⁻¹, at 1723cm⁻¹, at 1567cm⁻¹, at 1402cm⁻¹ [14] at 1334cm⁻¹ and at 1128cm⁻¹ [14-15].

The 2912cm⁻¹ ir peak was assigned to C-H stretching vibrations, probably from aliphatic -CH₂- groups in the polymer with carboxylate groups. The peak at 1723cm⁻¹ was assigned to the C=O stretching vibration of the carboxylic acid group. The peak at 1567cm⁻¹ was assigned to the asymmetric stretching of the carboxylate anion (COO⁻) group. The peak at 1402cm⁻¹ was assigned to the vibration C-O-H bending, in plane bend, of the carboxylate anion (COO⁻) group [14]. The peak at 1334cm⁻¹ was assigned to the C-H bending vibration, or to the C-O stretching when it was coupled to the C-H bending [14]. Two bands arising from C-O stretching, and O-H bending appeared in the spectra of carboxylic acids near 1320-1210cm⁻¹ and near 1440-1395cm⁻¹ respectively [14]. Both of these bands involve some interaction between C-O stretching and in-plane C-O-H bending [14]. The peak at 1128cm⁻¹ was assigned to the C-O stretching vibration associated with the C-O single bond in carboxylates.

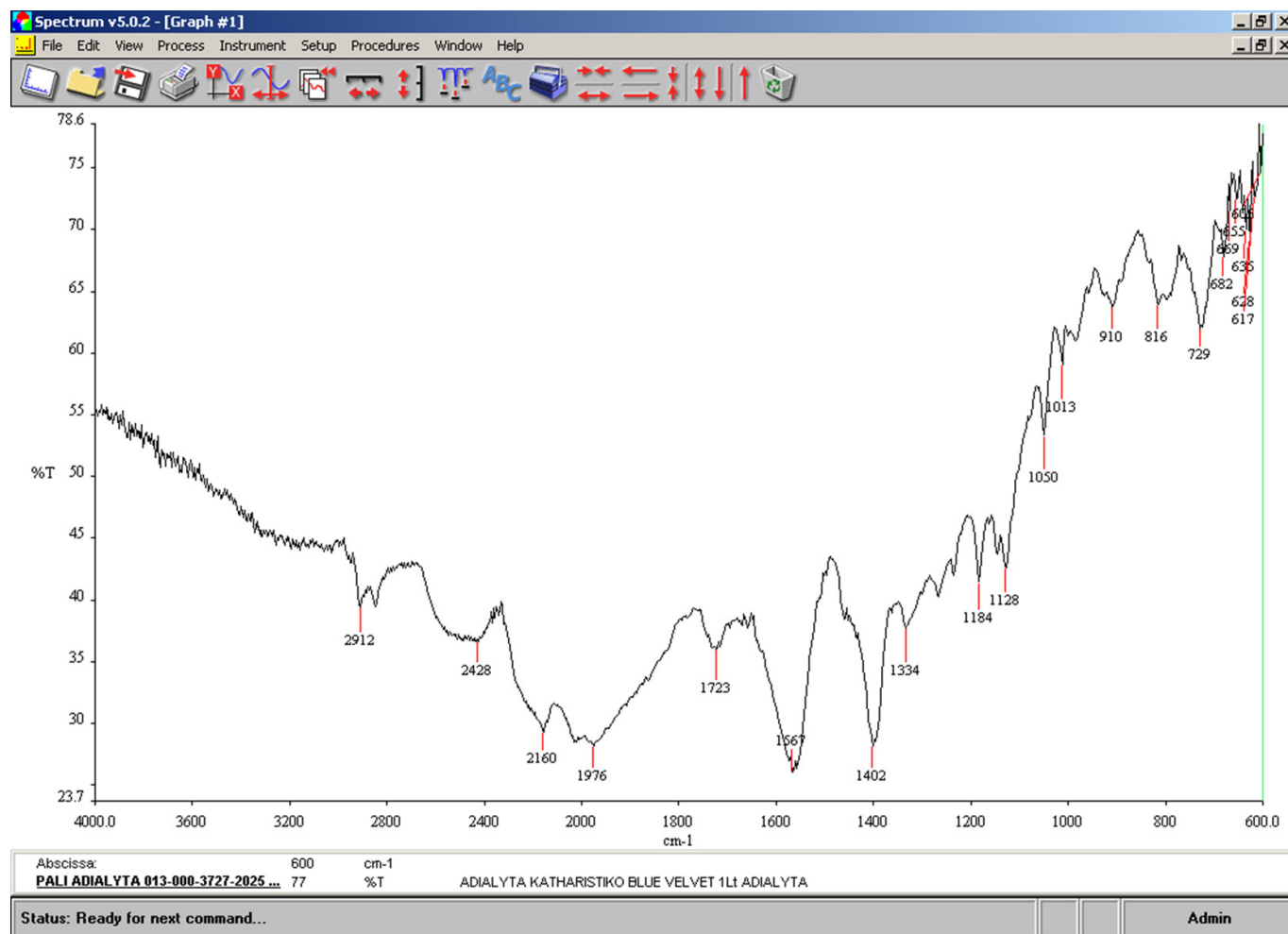


Figure 1: FT-IR spectrum of the insoluble matter ethanolic 95°, of the professional detergent product blue velvet 1L.

Also, the presence of the salt K_2CO_3 in the cleaning product blue velvet was identified from the ir peaks at 1050cm^{-1} , at 816cm^{-1} and at 669cm^{-1} . For the salt K_2CO_3 the peak at 1050cm^{-1} was assigned to the C-O stretching vibration of the carbonate ion [16]. The peak at 816cm^{-1} was assigned to the out of plane bending vibration of the CO_3^{2-} . The peak at 669cm^{-1} was assigned to the in-plane bending vibration of the carbonate ion [16].

In the spectrum in Figure 1, the ir peak at 2428cm^{-1} could be CO_2 in the sample, as the peak was assigned to an $O=C=O$ asymmetric stretch vibration [17]. The peak at 2160cm^{-1} was assigned to $C\equiv C$ alkyne stretch, was typical for $C\equiv C$ triple bond in conjugated systems present in the fragrance molecule [14-18]. The peak at 1976cm^{-1} was an overtone or combination band seen in carbonates structures [19]. The peak at 1184cm^{-1} was an -C-O-stretching vibration similar to -C-O-C- peaks which could be due to residues of the surfactant molecules in the mixture [20]. The peak at 1013cm^{-1} , may have been attributed to a C-O stretching vibration due to symmetric stretching in CO_3^{2-} carbonate ion [21]. The ir peak at 910cm^{-1} , was an =C-H out of plane bending from vinyl groups or alkenes and may have been due to degradation products of the detergent [22]. The peak at 729cm^{-1} was a rocking mode of long

chain (CH_2)-groups due to ordered alkyl chains from the surfactant molecule aggregates [23]. The peak at 682cm^{-1} could be associated to carbonate bending modes in the detergent product [24].

Secondly, we recorded the FTIR spectra of the soluble matter content ethanolic 95° (Figure 2), of the detergent product blue velvet 1L.

In Figure 2 the FT-IR spectrum of the soluble components in ethanol 95°, of the liquid cleaner was given, which was determined to be 3.15%w/w of the mixture of the detergent product blue velvet 1L. In the FT-IR spectrum in Figure 2, the anionic surfactant was identified at 2847cm^{-1} , at 1595cm^{-1} , at 1182cm^{-1} and at 1049cm^{-1} . The non-ionic surfactant was identified at 1121cm^{-1} .

The ir peak at 2847cm^{-1} was assigned to the C-H stretching vibration of the CH_2 groups $\nu_{\text{sym}}(CH_2)$ present in the alkyl chain of the hydrophobic tail of the anionic surfactant [25]. The peak at 1595cm^{-1} was assigned to the C=C stretching vibration of the benzene ring of the anionic surfactant [26]. The peak at 1460cm^{-1} could be attributed to the CH_2 bending vibration from the alkyl chain in the anionic surfactant present [25,26]. The ir peak at 1182cm^{-1} was assigned to the $S(=O)_2$ bond symmetric stretching vibration

from the sulfonate group of the anionic surfactant determined in the cleaner product [14,27]. The peak at 1049cm^{-1} was assigned to the S=O bond asymmetric stretching vibration for the sulfonate group [27]. The two peaks at 1182cm^{-1} and at 1049cm^{-1} supported the

presence of an alkyl sulfonate anionic surfactant in the detergent product blue velvet 1L, under study. The anionic surfactant was determined to be 0.52%w/w in the mixture of the product.

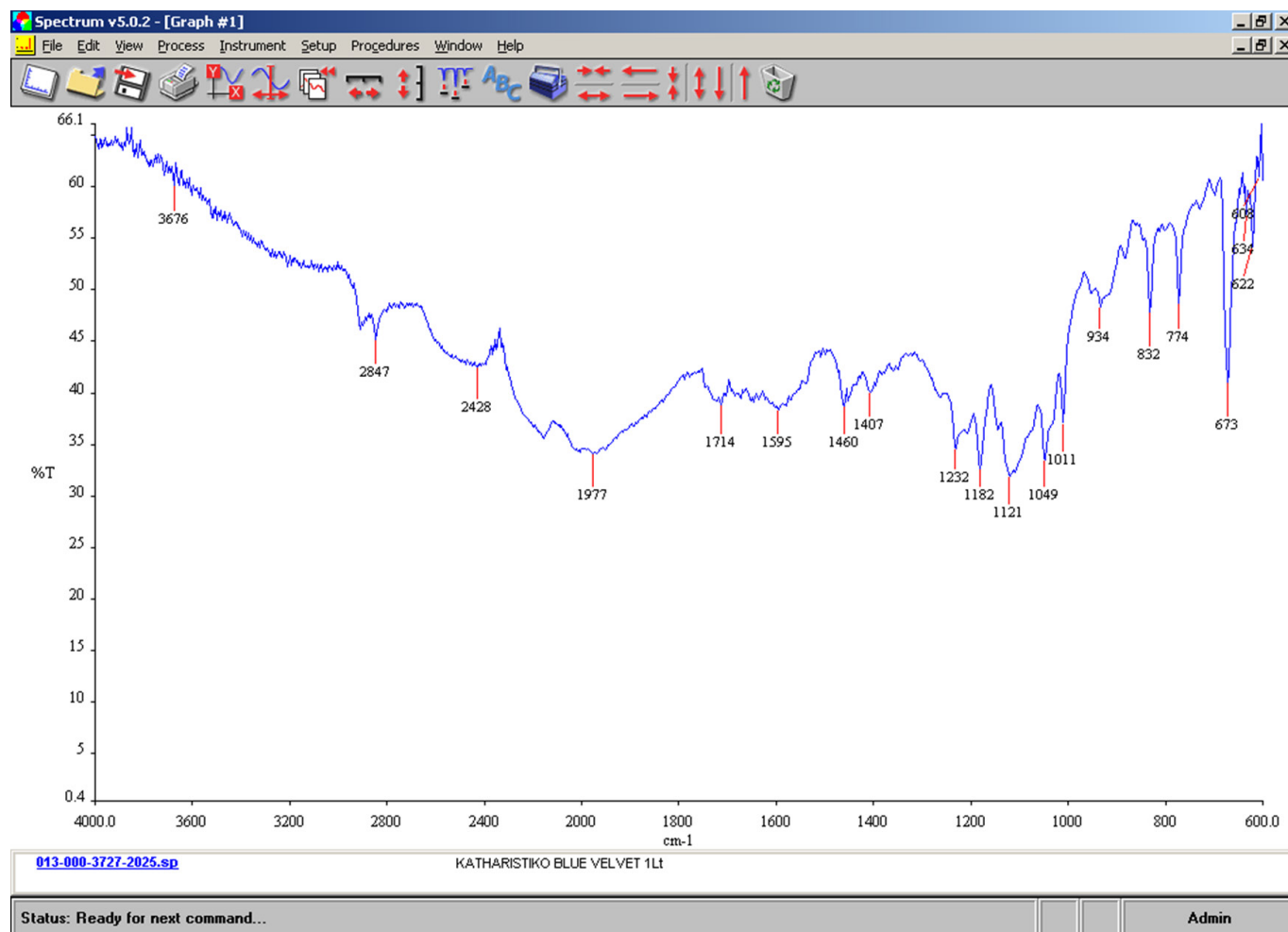


Figure 2: FT-IR spectrum of the soluble components in ethanol 95° of the professional detergent product blue velvet 1L.

The peak at 1121cm^{-1} was assigned to the CH_2 asymmetric stretching vibration of a non-ionic surfactant present in the mixture of the cleaning product [28,29]. Also, the sharp, weak peak at 3676cm^{-1} was assigned to the broad O-H stretching vibration indicative of a non-ionic surfactant with free terminal hydroxyl groups [30]. That peak was indicative of a free O-H group, which was non-hydrogen-bonded. The peak at 2847cm^{-1} was also assigned to the C-H symmetric stretching of the $-\text{CH}_2-$ groups present in the alkyl chain of the non-ionic surfactant present in the detergent product blue velvet 1L [25,31]. The ir peak at 1232cm^{-1} could be assigned to the S=O stretch of the sulfonate group (SO_3^-) of the anionic surfactant [25]. The non-ionic surfactant may have had many C-O-C bonds in its structure. So, the peak at 1011cm^{-1} corresponded to the $-\text{C}-\text{O}-$ stretching vibration within the ethoxylate or other polyether structure of the non-ionic surfactant [32]. The non-ionic surfactant could be determined to be approximately 2.11%w/w i.e. $(3.15\%w/w - 0.52\%w/w - 0.32\%w/w - 0.2\%w/w = 2.11\%w/w)$

present in the soluble components of the mixture of the product blue velvet 1L, whereas 3.15w/w was the soluble matter ethanolic, 0.52%w/w was the anionic-active matter content, 0.32%w/w was the base NaOH and 0.2%w/w was the fragrance of the cleaner product. The peak at 934cm^{-1} could be assigned to the $=\text{C}-\text{H}$ out of plane bending of unsaturated fragrance compounds of the cleaner product [33]. The peak at 832cm^{-1} corresponded to an aromatic ring C-H deformations [34]. The peak at 774cm^{-1} could be indicative of aromatic ring bending i.e. C-H out-of-plane bending vibration due to a benzene ring of the fragrance molecule in the product [35]. Also, the ir peak at 774cm^{-1} could be assigned also, to various strong S-O-C stretching vibrations, occurring in the region 1000cm^{-1} – 769cm^{-1} [14]. The strong peak at 673cm^{-1} could be attributed to CH_2 rocking (bending vibration), of a long alkyl chain due to the anionic surfactant of the cleaner product [36]. The peak at 634cm^{-1} could be assigned to the O-H out of plane bending vibration of the phenolic preservatives of the cleaner product [37]. The peak at

622cm⁻¹ could be a ring deformation and could be an aromatic ring vibration [38,39]. The ir peak at 608cm⁻¹ could be due to aromatic ring deformation due to fragrance bases (alcohol, OH out-of-plane bend) [40]. The ir peaks at 622cm⁻¹ and at 608cm⁻¹ could be due then to aromatic ring vibration, due to the fragrance bases.

Conclusion

In this work, we recorded the ir spectra of the soluble matter ethanolic 95° and the ir spectra of the insoluble matter ethanolic 95°. In the ir spectrum of insoluble components in ethanol we identified the presence of the poly-carboxylates chemical group which could be attributed to the 2912cm⁻¹, 1723cm⁻¹, 1567cm⁻¹, 1402cm⁻¹, 1334cm⁻¹ and 1128cm⁻¹ peaks. The anionic surfactant present could have been very likely a sulfonate because of the ir spectrum peaks of the soluble components in ethanol 95°, i.e. 2847cm⁻¹, 1595cm⁻¹ and primarily of the peaks 1182cm⁻¹ and 1049cm⁻¹. We have had surfactants present under basic conditions. The polycarboxylates detected may have acted as dispersants, as stabilizers and as chelating agents for hardness ions in the basic cleaning product [41,42]. Basic media, like in this detergent product, contained excellent nucleophiles and weak electrophiles [43]. The OH⁻ from the NaOH base product present in the cleaner product was a very strong nucleophile and a hard base. Also, the RO⁻ alkoxide ion could have been formed from the non-ionic surfactant's ethoxylate groups under the alkaline pH 11.73. Carboxylates RCOO⁻ from the polycarboxylic acid salts present would have been a strong nucleophile also and finally sulfonates (RSO₃⁻). The weak electrophiles would be ester groups present in the fragrance of the cleaner product. Also, weak electrophiles would have been carbonyls or carbocations formed during breakdown of some of the ingredients of the cleaner product.

So, a nucleophilic substitution reaction of the SN-2 type [44], could have taken place, as OH⁻ or RO⁻ may have attacked the weak electrophiles present in the mixture of the cleaning product. Also, a saponification reaction has also taken place in it, which means the base hydrolysis of esters [45]. The carboxylate anion from the soap, an anionic surfactant, reacted with the alcohol (OH⁻) [46].

Finally, the cleaning product already identified in its components by FT-IR, has undergone treatment, which preceded the determination of primary biodegradability with the confirmatory test, which was extraction with alcohol and separation first of the anionic surfactant by ion exchange. The alcohol extraction removed the inorganic components of the cleaner product which would have disturbed the biodegradability test [11].

In an analysis procedure, which combined FT-IR spectroscopy, which provided structural and functional group information, with liquid chromatography which separated and quantified individual components, a better understanding of the chemical composition and interactions within the liquid professional cleaning product have been obtained, which aided cleaning efficiency and environmental compatibility.

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