

# Synthesis of an unconventional cationic surfactant precursor

## Síntesis del precursor de un surfactante catiónico no convencional

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### Resumen

Este trabajo consiste en el desarrollo de un precursor de surfactante catiónico no convencional empleando el método estándar de síntesis de éteres de Williamson. Tratamos de diseñar un nuevo tipo de surfactantes heterogéminis con un espaciador rígido y grupos de cabeza no idénticos en la estructura del surfactante. En la síntesis sugerida establecemos el paso preliminar para la obtención un surfactante con dos anillos aromáticos como espaciador rígido. El precursor sintetizado proporciona a la estructura del surfactante un espaciador rígido debido a la presencia del grupo bifenilo, esperando que el surfactante presente una reducción tanto de la curvatura de los agregados micelares, así como de la concentración micelar crítica (CMC), en comparación con reportes previos, donde emplean surfactantes convencionales. Para establecer los pasos de la síntesis se varía la temperatura en el tiempo de reacción, así como la velocidad de adición del compuesto que contiene el grupo de cabeza, deseado que se pretenda añadir a la estructura del surfactante propuesto. Para la caracterización se emplearon las técnicas de espectroscopia de infrarrojo (FT-IR) y resonancia magnética nuclear (RMN).

**Palabras clave:** surfactante, síntesis, precursor, CMC.

### Abstract

The present research is about the development of a precursor of unconventional cationic surfactant by using the standard procedures of the Williamson ether synthesis. It has been intended to design a new type of heterogemini surfactant with a rigid spacer and non identical head groups in the structure of the surfactant. In the synthesis suggested, the preliminary step to obtain a surfactant structure with two aromatic rings as rigid spacer has been established. This synthesized precursor provides with a rigid spacer to the structure of the surfactant due to the presence of a biphenyl group; it is expected that, with this surfactant, it further presents a reduction on both the curvature of micellar aggregates as well as the Critical Micelle Concentration (CMC) in respect of those previous reports where conventional surfactants are used. To establish the steps of the synthesis, the temperature during the time of reaction has been varied, as well as the velocity of addition of the compound containing the head group which is pretended to be added to the surfactant structure proposed. For the characterization, the infrared spectroscopy technique (FT-IR) and the Nuclear Magnetic Resonance (NMR) were used.

**Keywords:** surfactants, synthesis, precursor, CMC.

### Introduction

Gemini surfactants are a new kind of surfactants. They have amphiphiles composed of two identical hydrophobic chains linked with a spacer moiety at the two head groups, (Menger *et al.*, 1991). Since they have amphiphilic behaviour and form micelles, surface properties of Gemini surfactants were studied and described in several reviews (Borse *et al.*, 2006). Many studies have shown that the spacer group has much effect on the properties of the solution in this kind of surfactants. In a comparison with ordinary monomeric surfactants, the CMC of the Gemini surfactants is considerably lower than ordinary surfactants.

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A Gemini surfactant with two hydrocarbon chains and two hydrophilic head groups in a molecule is commonly known for exhibiting the following unusual properties: a CMC lower than one or two orders of magnitude, more efficiency in lowering the surface tension of water, the properties of the theoretical bases at a relatively low concentration, comparing with monomeric and ordinary surfactants. Most Gemini surfactants have a symmetrical structure comprising identical hydrocarbon chain lengths and hydrophilic regions. Our current area of interest is a new generation of Gemini surfactants, which would have better properties and unique characteristics in the adsorption and micellization process. In recent research, carried out with Gemini surfactants in which the head groups were chemically non identical, showed good surface-active properties, such as low CMC and high efficiency in lowering the surface tension, and aggregation behavior in the solution.

A new family of materials based on unconventional structure of surfactants is undergoing an exploration in several laboratories. These surfactants present a different structure from the surfactants named geminis. The resulting materials can be self-assembled into both side-chain-like polymers (Edlund *et al.*, 1996). The most recent research on surfactants are focused in obtaining high-performance on areas of high-technology such as electronics, printing, magnetic recording, biotechnology and microelectronics (Lee *et al.*, 1995).

Although cationic surfactant complies with only a small portion of the market, its importance in practical applications continues to grow. It is used as antibacterial, liquid crystals, gene transfection agents, in road repairs, for reactions and in preparation of crystalline mesoporous materials (Esumi *et al.*, 1998).

Conventional surfactants molecules are generally composed of two parts: one polar head and one alkyl chain, incompatible with one another. They are known for their tendency to complete self-association and to develop super

molecular assemblies, called micelles. The formed micelles are of various types, shapes and sizes, such as globular, cylindrical and spherical (Maiti *et al.*, 2000). The characteristics of these aggregates are governed by the molecular structure and the conditions of the solution of the surfactant, in addition to physical parameters (Borse *et al.*, 2006). In recent years, several researchers designed newer molecular structures of surfactants with greater surface activity; these molecules were called bis-surfactants, later, they were called «gemini» surfactants (Menger *et al.*, 2000; Oda *et al.*, 2001). This kind of surfactants is considerably more surface active than the conventional surfactants called «monomeric» surfactants (Li *et al.*, 1991; Rosen *et al.*, 1994), the difference stems from the structure of a conventional surfactant has a single hydrophobic tail connected to an ionic group or polar head group, whereas a gemini surfactant has in a sequence a hydrocarbon chain, an ionic group, a spacer, a second ionic group and another hydrocarbon tail (Zana *et al.*, 1995; Menger *et al.*, 2001). The spacer can be formed of different natures such as short or flexible chains as methylene groups, rigid as stilbene, polar as polyether, and non polar as aliphatic or aromatic groups (Bunton *et al.*, 1971). The ionic groups can be positive as ammonium or negative as phosphate. The majority of geminis have symmetrical structures with two identical polar groups and two identical chains.

Moreover, some unsymmetrical geminis and geminis with three or more polar groups or tails have been recently reported. One type of these unsymmetrical geminis is the bis-quaternary surfactants with a general molecular formula  $C_nH_{2n+1}N+(CH_3)_2 - (CH_2)_m - N+(CH_3)_2C_nH_{2n+1} \cdot 2Br^-$  and are referred as m-s-m DMA (DMA = dimethyl ammonium bromide) surfactants (Srivastava *et al.*, 1998; Deacon *et al.*, 2003). These surfactants possess unique properties in their solution such as very low critical micellar concentration (CMC), high detergency, high solubilization and high surface wetting capability; these properties give them a wide range of applications in diverse areas

such as mining, petroleum, chemical, pharmaceutical industries, and biochemical research. They are also used as preservatives (Bakshi *et al.*, 2005), anticorrosive (Koopal *et al.*, 1995; Sharma *et al.*, 2005) and antimicrobial agents (Menger *et al.*, 1991).

In the early 1990s, (Menger *et al.*, 1991), the term gemini was assigned to these bis-surfactants that have a rigid spacer such as benzene or stilbene. The term was, then, extended to other bis or double tailed surfactants, irrespective of the nature of the spacer. Furthermore, it was examined the effect of the heterocyclic head group and the acetylenic spacer on the aggregation properties of cationic geminis (Menger *et al.*, 2000). Surfactants containing rigid hydrophobic groups are also called unconventional surfactants. The synthesis and behavior of these surfactants have been widely reported, however, the use of this new amphiphiles in the synthesis of polymers through heterogeneous polymerization techniques (emulsion or mini-emulsion) has been hardly studied (Zaragoza *et al.*, 2003). It has been recently reported that this surfactants can be used as potential gene delivery agents. Due to this application, the number of research on the molecular structure and their effect on solution of these surfactants have been increased today because these compounds offer an important tool for different fields as biotechnology, biochemistry, genetics, and molecular biology. The aim of this work and its originality establish the preliminary step of an unconventional cationic surfactant synthesis, with a rigid spacer structure provided from the molecule of bisphenol.

## Materials and Methods

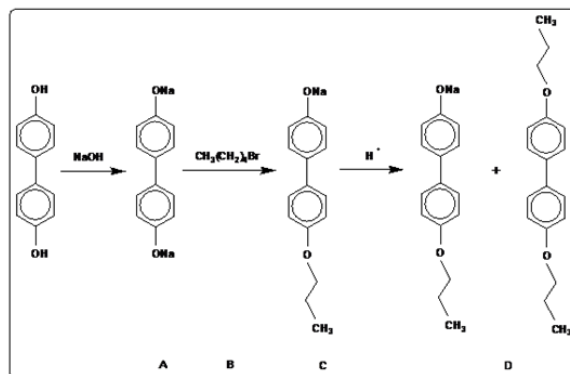
**Synthesis.** The precursor of this surfactant was prepared in two steps, by using the standard procedures of the Williamson ether synthesis (Brandys *et al.*, 1996). In the first step, the starting reactant, 4,4'-bisphenol 97% (Aldrich), was neutralized with two equivalent of 5% (p/v) sodium solution 3-8 mm spheres (Aldrich) in water. 1 equivalent of 1-bromopentane 99% (Aldrich) was dissolved separately in 99.5% ethanol and added

during the refluxing of the wise drop solution, and the refluxing continued for 72 h; the synthesis scheme is shown in Figure 1. The amount of products was registered after changing the time and temperature of reaction, as well as the velocity in which the compound contained the head group of the surfactant.

Most of the bi-substituted product was precipitated during this time and was collected after the mixture cooled at 2°C. The remaining solution contained most of the mono-substituted product. HCl concentrated was added to this solution, upon which an emulsion was formed. The emulsion was stirred for 30 min and then filtered and washed with water three times. The resulting white paste was dried at 80°C during three days. The dried paste was stirred for 20 min in chloroform and then filtered five times (during this step, the entire mono-substituted product was removed from the white paste), and re-crystallized.

In the second step, two equivalents of KOH reagent (Baker) were dissolved into dry methanol, to give a 5% (v/v) solution. The mono-substituted product was added, and a clear colorless solution was obtained on refluxing. An equivalent of 1,6-dibromopentane was added all at once to the refluxing solution. The refluxing continued for 72 h after overnight refluxing, the bi-substituted product precipitated; as a result, the volume of the mixture was reduced. The resulting precipitated product was collected and washed with 99.55% (v/v) of ethanol.

**Figure 1.** Scheme of unconventional cationic surfactant precursor synthesis. A, B, C and D are important steps carried out on the synthesis previously described.



**Analysis.** By using the infrared spectroscopy equipment Perkin Elmer brand GXATR model, infrared spectra were obtained. The <sup>1</sup>H NMR spectra were obtained by JNM GX400 FT-MNR.

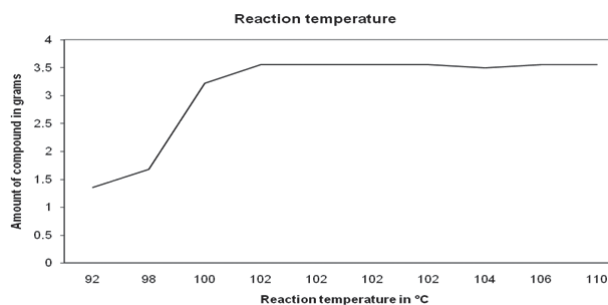
## Results and Discussion

**Synthesis.** To establish the optimum conditions to synthesize the mentioned surfactant, it was necessary to evaluate the effects of the reaction temperature, reaction time, and the adding velocity of the compound, which are going to give the head group to the structure of the surfactant. The analysis of these conditions was based on the impact of the amount of synthesized product. To analyze the effect of change in the conditions of the reaction, the amount of the obtained compound during the synthesis was registered and the interaction between the conditions was examined. The obtained data on the references suggested a starting temperature of 70°C, due to this result, the effect of the temperature was analyzed firstly (Bradys and Bazuin. 1998).

A summary of the results obtained in the experiments is shown below, only with the variation in the reaction temperature (Figure 2). The amount of compound did not change at higher temperatures than 120°C. However, at lower temperatures than 90°C, the amount of compound decreases significantly. This phenomenon could happen because at lower temperature it is more difficult for the molecules of bisphenol to be out of phase when a molecule of a reactant is attached, and more time available makes possible the substitution of the second -OH bond on the molecule, decreasing the amount of mono substituted compound, which is needed for the surfactant structure proposed.

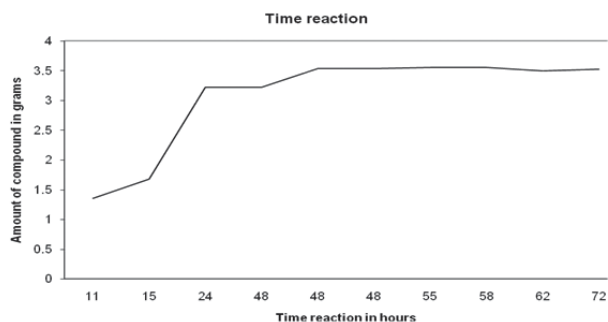
First of all, the effect of the temperature was analyzed as well as the reaction time, leaving constant the temperature at which the maximum of compound was obtained. The minimum time reaction was taken from references (Bradys and Bazuin. 1998).

**Figure 2.** Summary of the amount of compound obtained when the reaction temperature was varying.



Another experiments show the summary of the amount of compound in grams obtained when the time reaction was varying (Figure 3). If the reaction time is less than 48 h the amount of compound decreases and the un-reacted reactant amount increases. If the reaction time is more than 48 h the amount of compound does not change.

**Figure 3.** Summary of the amount of compound obtained when the time reaction was varying.

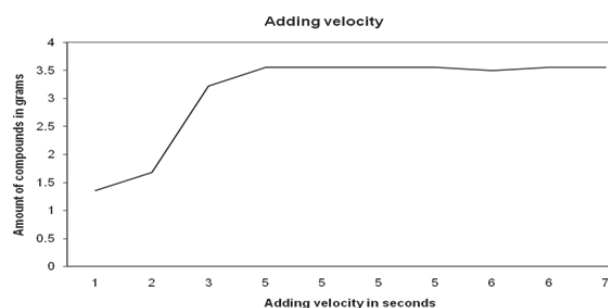


Through the bisphenol has two -OH bounds which can participate in the reaction and the main point is obtained the substitution of only one group, it was expected that decreasing the velocity in which the reactant is adding into the reaction the amount of mono-substituted compound will increase. For this, the adding velocity of the reactant was analyzed. It was found that, when the adding velocity decreases the amount of compound increases significantly. This might be, because the number of the molecules in the reactant is lower than the molecules of the bisphenol available, increasing the possibility of reaction of only one -OH bond with the reactant. On the other hand, if the adding



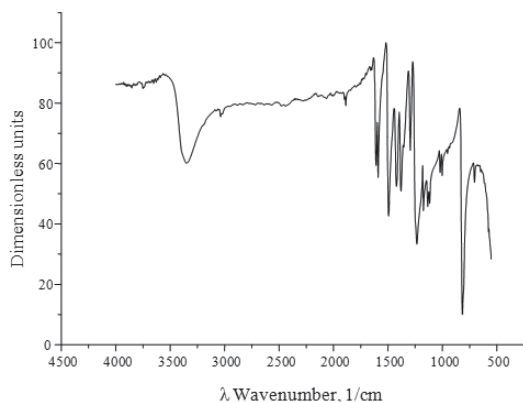
velocity increases the opposite effect is obtained, since the number of molecules in the reactant increases and the second -OH bond can be easily substituted (Figure 4).

**Figure 4.** Summary of the amount of compound obtained when the adding velocity was varying.



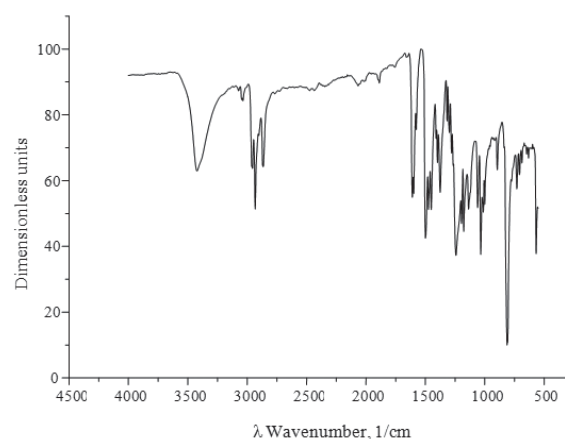
**Infrared Analysis.** The obtained products were filtered and dried to analyze them with infrared spectroscopy. The two important areas for preliminary analysis of infrared spectra are the regions at  $4000 - 1300$  and  $900 - 650$   $\text{cm}^{-1}$ . Figure 5 shows the spectra of the starting reactant. This spectrum has illustrated important infrared bands, which are very important for the synthesis. The non-hydrogen-bonded or free hydroxyl group of alcohols and phenols absorbs strongly around  $3500$   $\text{cm}^{-1}$  region. The C-O stretching vibrations in phenols produce a strong band in the  $1200 - 1000$   $\text{cm}^{-1}$  region of the spectrum. The C-O stretching mode is coupled with the adjacent C-C stretching vibration. The regions around  $1580$ ,  $1494$ ,  $1100$  and  $900$   $\text{cm}^{-1}$  are typically of bisphenols compounds.

**Figure 5.** Infrared spectra at room temperature of the starting reagent 4,4'-bisphenol.



The results during the infrared spectra of the substitute compound by only one hydroxyl group (Figure 6) show a region near to  $3300$   $\text{cm}^{-1}$  representing a free hydroxyl group of the molecule. Aromatic C-H stretching bands occur between  $3100$  and  $3000$   $\text{cm}^{-1}$ . Skeletal vibrations involving carbon-carbon stretching within the ring, absorb in the  $1600-1585$  and  $1500-1400$   $\text{cm}^{-1}$

**Figure 6.** Infrared spectra analyzed at room temperature of substitute compound by only one hydroxyl group.



The decrease of the intensity of the infrared spectrum around  $3300$   $\text{cm}^{-1}$  of the starting reactant, was caused by the substitution of a hydroxyl group of the molecule. Bands near to  $2930$   $\text{cm}^{-1}$  are attributed to bonds C-O-C of the nucleophilic substitution between the starting reactant and 1-bromoalcano.

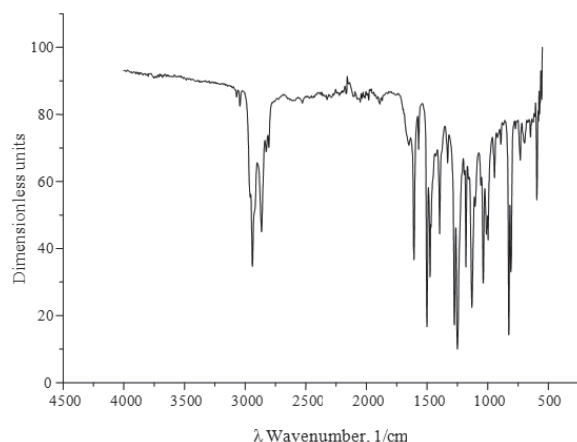
The infrared bands near to  $1494$   $\text{cm}^{-1}$  involve the next non-aromatic bonds: C-C and C-H. What is more, the region around  $809$   $\text{cm}^{-1}$  is caused by the substitution of the 4,4-bisphenol; the figure 2 shows a low intensity variation caused by these groups on the molecule. Finally, the appearance of the infrared band near to  $590$   $\text{cm}^{-1}$  involves stretching C-C and C-H bonds that confirm the substitution on the molecule of bisphenol bond to C-O. This suggest that the interaction of C-O bonding out-of-plane with C-C stretching and the C-H of the aliphatic chain as result of the substitution of the molecule of bisphenol, to analyze the spectrum regions near

to 2930, 1490, 809 and 509  $\text{cm}^{-1}$  reported (Zhao *et al.*, 2007). This experiment also shows the infrared spectra of the sub-product in the first part of the synthesis (Figure 6). In this spectrum it can be appreciated an increase in the intensity of the infrared band near to 2940  $\text{cm}^{-1}$  (Figure 3), in which the C-O-C stretching bonding was involved. This increase is caused by the elimination of the molecule of bisphenol free hydroxyl groups forming bonds C-O-C.

On the other hand, the increase in the intensity of the infrared band near to 1490  $\text{cm}^{-1}$  is attributed to the growing interaction between the C-C and the C-H bonds of the aliphatic chain with the molecule of bisphenol shown in Figure 5, through the elimination of another free hydroxyl group within the molecule.

Another region that support the bi-substitution of the molecule of bisphenol, is the appearance of the infrared bands near to 1034  $\text{cm}^{-1}$ , that involves stretching C-O-C bonding to asymmetric C-O of the compound. Furthermore, the intensity of the infrared band near to 825  $\text{cm}^{-1}$  is attributed to the bi-substitution of the 4,4-bisphenol for aliphatic chains, which intensity shows a low intensity due to the deference of the groups bonding to the molecule of bisphenol. To conclude, the increase of the infrared band near to 590  $\text{cm}^{-1}$  is attributed to the stretching of the C-C and the C-H of the aliphatic chains bonding to the C-O out-of-plane of the bi-substitute molecule.

**Figure 7.** Infrared spectra analyzed at room temperature of substitute compound, substitution of the free hydroxyl groups.



Infrared spectra in figure 7 are similar to infrared spectra in figure 3, due to the aliphatic chains in the molecule of bisphenol. The region near to 2900-2800 represents the stretching C-H of the aromatic ring, and typical stretching bands C-O-C of aril ester compounds. The band near to 1600  $\text{cm}^{-1}$  is attributed to the stretching C=C of the aromatic ring, infrared bands close to 1500  $\text{cm}^{-1}$  are attributed to aliphatic chains bonding to the compounds of bisphenol.

The region near to 1300-1200  $\text{cm}^{-1}$  shows the stretching C-O out-of-plane, this band increases its intensity shown in the spectrum of figure 3. Bands around 1100  $\text{cm}^{-1}$  are attributed to the stretching C-H in-the-plane of the aromatic ring, like in the infrared spectrum (Figure 3). The most important difference between the spectrum in figure 3 and the one shown in figure 4 is the decrease of the bands near to 700 – 500  $\text{cm}^{-1}$ , due to the difference of the aliphatic chain added to the molecule of bisphenol (Chorro *et al.*, 1998).

**NMR analysis.** The obtained products were filtered and re-crystallized twice with diluted ethanol, as a result, 1H NMR spectra by JNM GX400 FT-MNR was obtained.

**Figure 8.** 1H NMR spectra of the substitute compound by only one hydroxyl group.

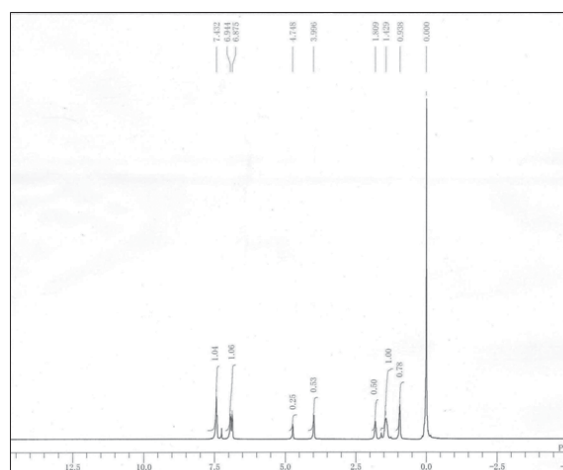


Figure 8 shows the 1H NMR spectra of the obtained compound during the first part on the

synthesis of the surfactant. These spectra show that the analyzed compound has the number of hydrogen expected for the molecule in the first part of the synthesis. This spectrum also shows that the substitutions of the carbons that form the chemical structure of the compound are also the expected on the structure of the substitute compound by only one hydroxyl group during the synthesis (Qiuging *et al.*, 2008).

## Conclusions

It is possible to obtain an unconventional cationic surfactant precursor with rigid spacer by the Williamson ether synthesis.

The reaction temperature has an important effect in the amount of precursor obtained. The most significantly effect of the synthesis was the variation of the adding velocity of the alkyl bromide, the maximum amount of precursor was obtained when the adding velocity decreased.

Due to the presence of hydroxyl groups in the molecule of bisphenol, the formation of bi-substitute product is easily observed. It is possible that the initial reagent molecule became out of phase in order to facilitate the reaction of one hydroxyl group, without the presence of several second reaction products, allowing the substitution on only one -OH bond in the bisphenol structure.


Regarding the summary, we conclude that the reaction conditions to obtain an 80% of product in the first part of the synthesis are a temperature of around 85°C, reaction time of 48 h and one drop added each 5 s. The reaction conditions to obtain 80% from the second part of the synthesis are a temperature above 100°C, and a reaction time of 48 h.

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**ALEJANDRO MARTÍNEZ MARTÍNEZ.** Tiene una amplia trayectoria en bioquímica y neurociencias. Es miembro de la Sociedad Mexicana de Bioquímica AC, y de la American Society of Neurosciences. En 1991 obtuvo la licenciatura en Biología en la Universidad de Guadalajara (UDG). Obtuvo el grado de Maestría en Neuroquímica en el Departamento de Química de la UNAM en 1994. En 1997, culminó sus estudios de Doctorado en Biología en la Universidad de Murcia, España. Realizó varias estancias académicas de posgrado, entre las que destacan su postdoctorado en la Universidad de California en San Diego (Irving), con una beca de la fundación hispana PEW, culminando en el 2003. El mismo año, ingresó como Profesor Investigador de tiempo completo a la Universidad Autónoma de Ciudad Juárez. Cuenta con múltiples publicaciones y capítulos de libros, así como dirección individual de tesis de pregrado y de grado. Imparte cátedra de ingeniería genética en el programa de química y bioinformática en la Maestría en Ciencias con orientación en genómica (PNP) y Maestría en Ciencias Químico- Biológicas (PNP).

**LUIS FERNANDO PLENGE TELLECHEA.** Desde 1990 ingresó como becario interno del laboratorio de reproducción en la Facultad de Ciencias de la Universidad Autónoma de Baja California. En 1992 culminó sus estudios de Biología en la misma dependencia. Posteriormente realizó sus estudios de Doctorado en Ciencias Biológicas por la Universidad de Murcia, culminando en 1998. El Dr. Plenge se ha caracterizado por sus estudios bioquímicos en la rama de proteínas asociadas en membranas. Actualmente labora como profesor investigador de tiempo completo en la Universidad Autónoma de Ciudad Juárez. Cuenta con múltiples publicaciones y dirección de tesis de pregrado y de grado. Es actual director en jefe de la revista de ciencias, *Ciencia en la Frontera*, e imparte la cátedra de Bioquímica en el programa de Biología y de Estructura y función proteínas en la Maestría en Ciencias con orientación en genómica (PNP). Actualmente se encuentra en una estancia Posdoctoral en el Border Biomedical Research de la Universidad de Texas en El Paso desde 2010 al presente, estudiando mecanismos bioquímicos de neurotransportadores.

**TAKAOMI KOBAYASHI.** Miembro del Departamento de Química de la Nagaoka University of Technology. Ha recibido el premio al mejor investigador joven en polímeros de Japón, actualmente es presidente del Consorcio JAP-MEX-USA. Su área de investigación es el desarrollo de materiales poliméricos. Entre los logros más recientes de su equipo de colaboradores es el desarrollo de una nueva técnica de impresión molecular en la preparación de membranas porosas.