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Study on interaction between palladium(II)–Linezolid chelate with eosin by resonance Rayleigh scattering, second order of scattering and frequency doubling scattering methods using Taguchi orthogonal array design

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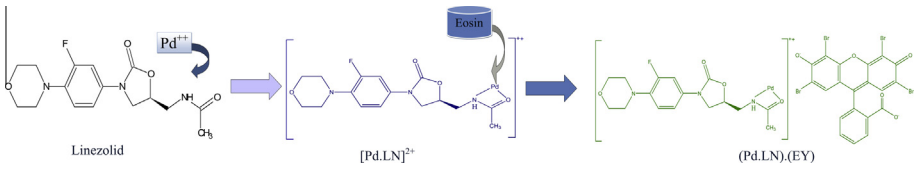
HIGHLIGHTS

- Three new sensitive scattering methods were developed to determine Linezolid.
- Linezolid reacted with Pd(II) and eosin to form 1:1:1 ternary ion association complex.
- Optimizations of method parameters are done using Taguchi orthogonal array design.

GRAPHICAL ABSTRACT



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ABSTRACT

Linezolid reacted with palladium to form 1:1 binary cationic chelate which further reacted with eosin dye to form 1:1 ternary ion association complex at pH 4 of Walpole's acetate buffer in the presence of methyl cellulose. As a result not only absorption spectra were changed but Resonance Rayleigh Scattering (RRS), Second-order Scattering (SOS) and Frequency Doubling Scattering (FDS) intensities were greatly enhanced. The analytical wavelengths of RRS, SOS and FDS (λex/λem) of ternary complex were located at 538 nm/538 nm, 240 nm/480 nm and 660 nm/330 nm, respectively. The linearity range for RRS, SOS and FDS methods were 0.01–0.5 μg mL^{-1}, 0.1–2 μg mL^{-1} and 0.2–1.8 μg mL^{-1}, respectively. The sensitivity order of three methods was as RRS > SOS > FDS. Accuracy of all methods were determined by recovery studies and showed recovery between 98% and 102%. Intraday and inter day precision were checked for all methods and %RSD was found to be less than 2 for all methods. The effects of foreign substances were tested on RRS method and it showed the method had good selectivity. For optimization of process parameter, Taguchi orthogonal array design L8(2^4) was used and ANOVA was adopted to determine the statistically significant control factors that affect the scattering intensities of methods. The reaction mechanism, composition of ternary ion association complex and reasons for scattering intensity enhancement was discussed in this work.

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Introduction

Linezolid (S)-N-({3-[3-flouro-4-(morpholin-4-yl)phenyl]-2-oxo-1, 3-oxazolidin-5-yl}methyl) acetamide; is a synthetic antibiotic belonging to a new class of antimicrobial called the oxazolidinones. It is active against most gram positive bacteria including methicillin resistant *Staphylococcus aureus* (MRSA), *Streptococcus pneumonia*, and vancomycin resistant *Enterococci* spp. strains. It acts by inhibiting initiation of bacterial protein synthesis. It may be particularly useful as an alternative to vancomycin in patients with impaired renal functions, in cases of patients with poor or no intravenous access and in patient who require outpatient therapy or unable to tolerate glycoside [1–3].

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At present, methods in literature reported for determination of Linezolid consist of HPLC [4,5], capillary electrophoresis [6,7], stability indicating liquid chromatography for enantiomeric separation of Linezolid, spectrophotometry [8] etc. However, there are no research work has been reported for determination of Linezolid by RRS, SOS and FDS methods.

As a new analytical techniques RRS, SOS and FDS have been drawn more attention because of their higher sensitivities and simplicities [9]. These techniques have been applied to determine macromolecules such as proteins [10–12], polysaccharides [13–16], nucleic acids [17,18], trace metal ions [19,20], surfactants [21,22] and pharmaceuticals [23–26].

In our experiments, we found that in pH-4 Walpole's acetate buffer, at room temperature, palladium reacted with Linezolid to form 1:1 cationic chelate $[Pd{\cdot}LN]^{+2}$, which further reacted with eosin dye to form 1:1 ternary ion association complex $[Pd{\cdot}LN]{\cdot}(EY)$, in presence of methyl cellulose. Due to formation of ternary ion association complex intensities of RRS, SOS and FDS were greatly enhanced. The increments of intensity (ΔI) were directly proportional to concentration of Linezolid in certain ranges.

For optimization of process parameter, in former all studies of involving RRS, SOS and FDS used the one factor at one time strategy [27–30]. This strategy involves changing one at a time, while leaving the others factors unaltered. The one factor at a time requires more experimental trials. The advantage Taguchi orthogonal array design is that numerous factors can be simultaneously optimized by using few experimental trials. The main advantage of Taguchi orthogonal array design over the conventional statistical design is that, conventional statistical design can determine the optimum condition on the basis of the measured values of characteristic properties, while Taguchi orthogonal array design does this on the basis of variability of characteristic properties. In other words, the Taguchi orthogonal array design can determine the optimum experimental condition having least variability in results [31–36]. Because of that, in this work, we used Taguchi orthogonal array design $L8(2^4)$ with ANOVA to study effects of control factors on RRS intensity. Among the all three methods RRS method had the highest sensitivity, so that optimization by using Taguchi design was applied for RRS method only. The effects of foreign substances were investigated and results showed that RRS method had good selectivity. Therefore, all three methods can be applied for determination of Linezolid in human plasma. In addition, reaction mechanism and reasons for scattering intensity enhancement were discussed.

Experimental

Instruments

A Shimadzu RF-5301PC spectrofluorophotometer (Tokyo, Japan), equipped with 150 W xenon lamp, was used to record RRS, SOS and FDS spectra. A UV-1700 double beam Spectrophotometer (Tokyo, Japan) was used for recording absorption spectra.

Reagents and chemicals

All reagents were of analytical grade and double distilled water was used throughout work.

Linezolid stock solution: Accurately weighed 5 mg of Linezolid (obtained from Cipla Pvt. Ltd., India) was transferred to 50 ml volumetric flask and make up the volume with methanol to obtain stock solution of 100 μg mL^{-1}. From these stock solution Linezolid (10 μg mL^{-1}) were prepared by transferring 2.5 ml in 25 ml volumetric flask and make up volume with water.

Eosin stock solution: Eosin (Sigma Aldrich, India) was prepared as 2×10^{-3} M solution by dissolving accurately weighed 69.15 mg of eosin in water and working solution of 2×10^{-5} M was prepared by further dilution with water.

Palladium acetate solution: Stock solution of 2×10^{-4} M was prepared by dissolving 11.22 mg of palladium acetate (Sigma Aldrich, India) in 2 ml of concentrated hydrochloric acid followed by addition of 60 ml of boiled water and diluting to 100 ml with water. From this working solution of 2×10^{-5} M was prepared by transferring suitable aliquots.

Methyl cellulose solution: 0.5% w/v solution of methyl cellulose was prepared by dissolving 250 mg of methyl cellulose in appropriate amount of hot water (80 °C) with stirring for 20 min and then chilling to 5 °C in freeze for 15 min.

Walpole's acetate buffer: It was prepared by adding 0.2 M sodium acetate (Allied chemical Co., India) in 0.2 M acetic acid (Fisher Scientific Pvt. Ltd., India) until pH-4 was obtained.

Taguchi orthogonal array design

In Taguchi design, the primary goal is to find factor setting that minimizes response variation, while adjusting process on target. After determination of factors which affect variation, then try to find out setting for controllable factors that will either reduce the variation or make the procedure insensitive to changes in noise factor or both. A procedure design with this goal will give more consistent performance regardless of the environment in which it is used.

In this work, four controlling factors (independent factor) were considered in affecting scattering intensities based on analysis of our laboratory scale operation. This four controlling factors are concentration of eosin (A), concentration of palladium acetate (B), pH (C) and concentration of methyl cellulose (D). For each control factor two different levels are selected. The selected ranges between two levels depend on prior laboratory scale optimization. So in this work, we use 0.7 ml (level 1) and 0.75 ml (level 2) for eosin of 2×10^{-5} M and palladium acetate of 2×10^{-5} M. pH 3.8 and pH 4 were respectively selected as level 1 and level 2 and for 0.5% methyl cellulose 1.4 ml (level 1) and 1.5 ml (level 2) were used. For this four control factors eight test conditions were designed according to $L8(2^4)$ orthogonal array.

General procedure

A suitable amount of Linezolid was pipetted into 10 ml volumetric flask followed by 1 ml of pH-4 Walpole's acetate buffer, 1.5 ml of methyl cellulose solution, 0.75 ml of eosin solution, 0.75 ml of palladium acetate solution and make up the volume with water and shake thoroughly. Then set aside for 20 min. The RRS intensity of system were recorded with synchronous scanning at λem = λex. The SOS intensity and FDS intensity of system were recorded at λem = 2λex and 2λem = λex, respectively. The I_{SOS} and I_{FDS} were plotted versus different wavelength to obtain excitation and emission wavelength for SOS and FDS methods, respectively. Then, the scattering intensities ΔI_{RRS}, ΔI_{SOS} and ΔI_{FDS} for ternary ion association complex were measured by subtracting actual intensities with reagent blank at their own analytical wavelength.

Result and discussion

Spectral characteristics

RRS spectra

The RRS spectra of $[Pd{\cdot}LN]{\cdot}(EY)$ system were recorded by synchronous scanning from 220 nm to 770 nm. The results are shown in Fig. 1. From where, it can be seen that RRS intensities of blank

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