



# An environmentally friendly method to remove and utilize the highly toxic strychnine in other products based on proton-transfer complexation

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## ARTICLE INFO

### Article history:

Received 17 May 2015

Received in revised form

26 August 2015

Accepted 29 August 2015

Available online 3 September 2015

### Keywords:

Strychnine

Proton-transfer

Nanoporous carbon

SEM

TEM

## ABSTRACT

The study of toxic and carcinogenic substances represents one of the most demanding areas in human safety, due to their repercussions for public health. There is great motivation to remove and utilize these substances in other products instead of leaving them contaminate the environment. One potentially toxic compound for humans is strychnine (Sy). In the present study, we attempted to establish a quick, simple, direct and efficient method to remove and utilize discarded Sy in other products based on proton-transfer complexation. First, Sy was reacted with the acido organic acceptors PA, DNBA and CLA. Then, the resultant salts were direct carbonized into carbon materials. Also, this study provides an insight into the structure and morphology of the obtained products by a range of physicochemical techniques, such as UV–visible, IR, <sup>1</sup>H NMR and <sup>13</sup>C NMR spectroscopies; XRD; SEM; TEM; and elemental and thermal analyses. Interestingly, the complexation of Sy with the PA or DNBA acceptor leads to a porous carbon material, while its complexation with CLA acceptor forms non-porous carbon product.

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## 1. Introduction

Strychnine (Sy), chemically strychnidin-10-one, the structure of which is shown in Fig. 1, is a monoterpene indole alkaloid found as a white odorless and bitter crystalline powder. The primary natural source of Sy is the seeds of *Strychnos nux-vomica* L. (Loganiaceae), a tree grown extensively in China and southern Asian countries [1,2]. It is found together with brucine and other indole alkaloids in various plants of the strychnine family [2]. It has been effectively used in traditional Chinese medicine to treat central nervous system diseases and to alleviate allergic symptoms, joint pain and traumatic pain [3]. Sy when ingested could stimulate the central nervous system and make the sensory organs more sensitive [4]. So at low doses (such as at 10 mg daily dose) it is often used to treat nervous diseases and vomiting as well as arthritic and traumatic pains [5]. However, Sy is highly toxic to humans and most domestic

animals and the margin between therapeutic and toxic doses is very narrow as it was reported to be fatal to man at doses of 30–90 mg [6]. It has been demonstrated that a high of the Sy can induce convulsions of the central nervous system and death through respiratory or spinal paralysis or cardiac arrest [7]. Nowadays, Sy is no longer used as a therapeutic drug and its availability to the public is controlled by legislations in various jurisdictions, but it is still in limited use as a rodenticide, pesticide, fungicide and an adulterant in street drugs [8,9]. Porous and nanostructured carbon materials have attracted much attention because of their versatile applications in catalysis, sensors, electronic devices, electrodes, gas and liquid separation, and memory storage [10–12]. Activated porous carbons have received significant attention as potential hydrogen storage media due to their low density, high surface area, large pore volume, good chemical stability, and high storage capacity [13]. For several years, we have investigated the synthesis, characterization and application of various charge-transfer (CT) and proton-transfer (PT) interactions [14–28]. As part of our continuing interest in this field, in this work, we originally report a method to remove and utilize discarded Sy in

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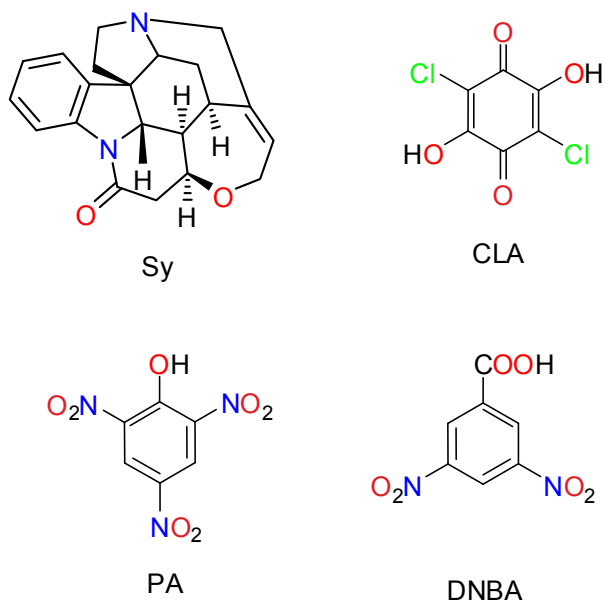


Fig. 1. The structure of strychnine (Sy) and organic acceptors.

other products as porous carbon material. First, Sy was directly reacted with acido organic acceptors (PA, DNBA and CLA) to form proton-transfer salts. Then, the resultant salts were direct carbonized at 400 °C into carbon materials. The obtained products were characterized by elemental, thermal and spectroscopic data (UV–Vis, IR,  $^1\text{H}$  and  $^{13}\text{C}$  NMR). Their morphology and nanometry were observed and differentiated using X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques.

## 2. Experiment and calculations

### 2.1. Chemicals and solutions

All of the chemicals used were of analytical grade and were used as purchased. Strychnine (Sy;  $\text{C}_{21}\text{H}_{22}\text{N}_2\text{O}_2$ , 334.42) was supplied by Sigma–Aldrich Chemical Co. (USA). The organic acceptors picric acid (PA;  $\text{C}_6\text{H}_3\text{N}_3\text{O}_7$ ; 229.10), 3,5-dinitrobenzoic acid (DNBA;  $\text{C}_7\text{H}_4\text{O}_6\text{N}_2$ ; 212.12), and chloranilic acid (CLA;  $\text{C}_6\text{H}_2\text{Cl}_2\text{O}_4$ ; 208.98) were purchased from Merck (Darmstadt, Germany) and were used without modification. HPLC-grade methanol and chloroform were also purchased from Merck. Standard stock solutions at a concentration of  $5.0 \times 10^{-3}$  M were freshly prepared prior to each series of measurements by dissolving precisely weighed quantities in a 100 mL volumetric flask. The stock solutions were protected from light. Solutions for spectroscopic measurements were prepared by mixing appropriate volumes of the Sy and acceptor stock solutions with the solvent immediately before recording the spectra.

### 2.2. Characterization methods

#### 2.2.1. CHN analysis

To ascertain the constituents, purity and compositions of the synthesized salts, the carbon, hydrogen and nitrogen contents were analyzed with a Perkin–Elmer 2400 series CHN microanalyzer (USA).

#### 2.2.2. UV–Vis spectra

The UV–Vis spectra were recorded over a wavelength range of

200–800 nm using a Perkin–Elmer Lambda 25 UV/Vis double-beam spectrophotometer with quartz cells. The path length of the cells was 1.0 cm.

#### 2.2.3. IR spectra

The infrared (IR) spectra of the solid products (as KBr discs) were acquired at room temperature using a Shimadzu FT-IR spectrophotometer (Japan) over the range of 4000–400  $\text{cm}^{-1}$ .

#### 2.2.4. $^1\text{H}$ and $^{13}\text{C}$ spectra

$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were collected on a Bruker DRX-250 spectrometer operating at 600 MHz. The measurements were performed at ambient temperature using  $\text{DMSO-d}_6$  (dimethylsulfoxide,  $\text{d}_6$ ) as the solvent and TMS (tetramethylsilane) as the internal reference.

#### 2.2.5. Thermal analysis

Thermogravimetric (TG) analysis was performed using a Shimadzu TGA–50H thermal analyzer (Japan) with standard platinum TG pans. The measurements were conducted at a constant heating rate of 10 °C/min over the temperature range of 25–600 °C in a nitrogen atmosphere using alumina powder as the reference material.

#### 2.2.6. XRD analysis

The X-ray diffraction (XRD) profiles were obtained using a PANalytical X'Pert PRO X-ray powder diffractometer. The instrument was equipped with a Ge(III) secondary monochromator, and  $\text{Cu K}\alpha_1$  was employed as the radiation source, with a wavelength of 0.154056 nm. The samples were scanned with  $2\theta$  between 5° and 90°.

#### 2.2.7. SEM analysis

The microstructure and morphology were analyzed by a scanning electron microscope (SEM, Quanta FEG 250 instrument). The instrument was operated at an accelerating voltage of 20 kV.

#### 2.2.8. TEM analysis

The particle size was analyzed by transmission electron microscope (TEM, JEOL JEM-1200 EX II, Japan). The instrument was operated at an accelerating voltage of 60–70 kV.

### 2.3. Preparation of materials

#### 2.3.1. Preparation of the salts

A simple synthetic protocol has been used for the preparation of PT complexes of Sy donor. A typical procedure for the preparation is briefly described as follows. First, 2 mmol of Sy in chloroform (20 ml) was added to 20 ml of a solution containing 2 mmol of the acceptor (either PA, DNBA or CLA) in the methanol solvent. The resulting mixture was stirred at room temperature for approximately half an hour, where the resulting precipitation were isolated as yellow canary, white and reddish-brown powder for Sy–PA, Sy–DNBA and Sy–CLA salts, respectively. The formed products were isolated, filtered and further purified using methanol-chloroform solvent and a recrystallization process to obtain the pure products. The products were then collected and dried *in vacuo* for 48 h. The products were characterized by spectroscopy (UV–Vis, IR,  $^1\text{H}$  and  $^{13}\text{C}$  NMR) as well as elemental and thermal analyses. The excellent agreement between the experimental and calculated values of C, H and N indicates that the obtained products are free of impurities. The stoichiometry of the Sy interaction with the acceptors was found to have a 1:1 ratio.

2.3.1.1. Sy free. white powder; IR (KBr,  $\text{cm}^{-1}$ ):  $\nu_{\text{max}}$  3050–2950

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