



Higher methane storage at low pressure and room temperature in new easily scalable large-scale production activated carbon for static and vehicular applications

Alfonso Policicchio^{a,*}, Enrico Maccallini^a, Raffaele Giuseppe Agostino^a, Federica Ciuchi^b, Alfredo Aloise^c, Girolamo Giordano^c

^a Dipartimento di Fisica, Università della Calabria, Via Ponte P. Bucci, Cubo 31C, 87036 Arcavacata di Rende (CS), Italy

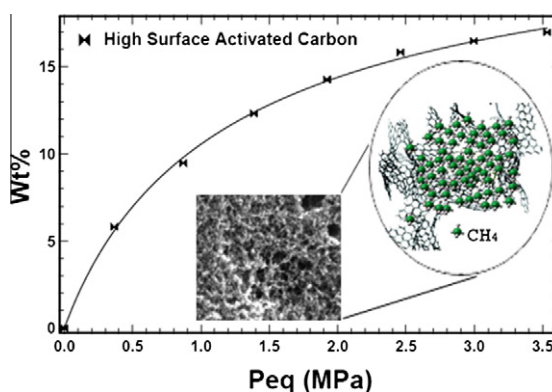
^b CNR – IPCF UOS Cosenza LiCryL, c/o Università della Calabria, Via Ponte P. Bucci, Cubo 31C, 87036 Arcavacata di Rende (CS), Italy

^c Dipartimento Ingegneria Chimica e dei Materiali, Università della Calabria, Via Ponte P. Bucci, Cubo 42A, 87036 Arcavacata di Rende (CS), Italy

HIGHLIGHTS

- ▶ High Surface Activated Carbon for methane physisorption.
- ▶ Fully reversible methane storage at ambient condition.
- ▶ Simple, cheap and effective synthesis process.
- ▶ Suitable for automotive application.

GRAPHICAL ABSTRACT



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ABSTRACT

The methane adsorption properties of High Surface Activated Carbon (HSAC) samples were evaluated at room temperature (298 K) and pressure up to 3.5 MPa using a new optimized volumetric apparatus *f*-PcT (fast Pressure–concentration–Temperature) for accurate and reliable gas adsorption measurements. A comprehensive characterization of different activated carbon samples was carried out by means of helium pycnometry for the skeletal density evaluation, by the Brunauer–Emmett–Teller (BET) method for the measurement of the surface area, by Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) for topography and long-range order estimation, respectively.

All the adsorption data were evaluated by Langmuir/Tóth isotherm model with a very high accuracy. Comparison with data available in the literature shows a good agreement in terms of maximum methane uptake on similar materials and an enhanced performance for the reversible adsorption at very low pressure. In fact, the probed HSAC samples show both higher methane storage values for pressure up to 1.5 MPa and totally reversible methane uptake up to many cycles without any treatment in between. Furthermore, their adsorption properties are stable also after air exposure.

These results represent the starting point for a real and efficient alternative method to the natural gas storage for static and/or automotive applications.

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1. Introduction

Over the past 10 years, the major governments are implementing important policies to support renewable energy and environ-

* Corresponding author. Tel.: +39 0984 49 6095; fax: +39 0984 49 4401.

E-mail address: alfonso.policicchio@fis.unical.it (A. Policicchio).

mentally sustainable energy. This new position, which arises from both an economic necessity and from an increasing awareness of environmental considerations, has directed and led the scientific community and industry to research and develop new and effective alternatives to low fuel environmental impact.

All these reasons, together with the growing demand for electric power and transportation fuels pushed the EU and US governments [1,2] and science [3] to look for new and clean energy-carrier resources.

Till now the clean and renewable energy, though promising, have played only a small part in today's energy picture [4] and it has to be further developed before it can play a much larger part in the future of the power and transportation sectors to reach the current level of needs.

The discovery of abundant natural gas reserves in several parts of the world, makes them the dominant source of energy for both sectors [5] in the incoming decades.

Natural gas in fact, is considered as an appropriate alternative fuel due to its huge resources and, because of its low price and low toxic gas emission and because it is also renewable from biomass and refuse [4], it is becoming economically attractive for many industrial applications around the world especially for vehicular uses to cover all energy demand [6,7].

However, a large fraction of natural gas is methane (CH_4) which has a low volumetric energy density, hence its use is limited because of storage and transportation problems. That is the reason why its compression and storage are of great interest to industrial and scientific communities.

The main challenge is to store natural gas close to room conditions (temperature and pressure) to reduce transportation limitations and to facilitate its use [6].

In view of this important goal, great care must be taken into account in the evaluation of the methane storage capacity which should reach, for the year 2015, a target of 150 v/v [8], which has been recently revised to 180 v/v [9–11] (the volume of gas adsorbed at standard temperature and pressure: 298 K, 0.1 MPa, per volume of the storage vessel) or equivalently 35 wt% [12] at 3.5 MPa according to U.S. Department of Energy (DOE).

Nowadays, the current methods for methane storage involves its compression into tanks at high pressure (CNG) [13,5] (in this case tanks mass gives a big contribution to the storage system mass decreasing significantly the methane wt% capacity of the whole system) and/or storage in the liquid form (LNG) at temperatures down to 120 K (mainly used for intercontinental transportation).

Together with the energy density requirements, methane portability has strict prescription in terms of safety, refilling and delivery performances. In this perspective, studies of adsorbed natural gas (ANG) and natural gas hydrate (NGH) have been carried out aiming to represent an efficient alternative solution for CH_4 portability [4].

The latter is solid and has a theoretical volumetric capacity of 164 or 174 v/v assuming complete filling of the water cages within the clathrate structure, but the formation condition of NGH is rigorous and its formation rate is low [4]. In addition, the stored gas cannot be released from hydrate just by reducing pressure, and hence it is not suitable as commercial technique for methane storage.

Whereas all, compared to pressurized gaseous, cryogenically liquefied methane or any other storage method actually on the market, methane storage into the solid materials (ANG) presents advantages according to gravimetric and volumetric energy density, safety, and energy efficiency. It is important to note that critical issues in the commercialization of adsorbed natural gas storage technology not only include the storage capability of the adsorbent but also the facility of use and the cost of manufacturing.

The most important evaluation of an ANG storage systems performance is the measurement of the volume of usable gas [14]. This is what scientists frequently called *delivery*, and it is defined as the volume of gas obtained from the storage vessel when the pressure is reduced from the storage pressure of 35–1 bar, at room temperature [14].

Despite everything the ANG technology in which natural gas is adsorbed by a porous adsorbent material at relatively low pressures (35/40 bar) has been considered like a promising solution in energy storage and especially in natural-gas fuelled vehicles sectors [15].

Within this global energy scenario it evident that one of the most important goal to extensively use of methane is the synthesis of methane storage materials with good adsorption properties under convenient temperature and pressure, but in particular with on-board operating storage capabilities for vehicular applications [1,11].

Up to now, all kind of available materials present different limitations especially for on-board storage applications: (1) relatively high thermal stability, (2) slow desorption/absorption kinetics, (3) unstable structures [16], (4) high weight, (5) irreversibility on cycling [4] and (6) expensive production costs. For these reasons research has been focused to the development and to the analysis of meso- and nano-structured system with high specific surface area to exploit the methane physisorption.

Among adsorbents investigated, carbon materials are the most effective in storage of natural gas at low pressure (e.g. 3.5/4.0 MPa).

Large number of paper have been published about gas adsorption at room temperatures on porous solid for its use as vehicular fuel [6]. Many porous solid are tested as an adsorbent for gas storage such as inorganic materials, zeolithes, silica gels, activated carbon. Ailing Cheng and Wun-Liang Hang choose activated carbon as the best adsorbent compared to porous clay [6].

Activated carbon can be based on anthracites, petroleum wastes or vegetal raw materials to be chosen as an important gas adsorbent. Precursors can be different types of vegetal grains such as olive stones [17], novel corn grain [18], anthracites [19].

Naturally, biomasses sources would be highly desirable of favorable to be converted into activated carbon used as the most important adsorbent for gas storage.

The delivery of gas from an activated carbon depends mainly on micropore volume, micropore size distribution and packing density of the activated carbon [5–7,20,21]. In this application, where the storage volume available is a constraint, increasing the packing density of the adsorbent in order to increase the ANG energy density is very important [15].

Experiments [22] were done also on the enhancement effect of pre-adsorbed water on methane storage on activated carbon. Adsorption isotherms were measured at 275–283 K and pressures up to 11 MPa but a significant increment in the methane adsorption is observed only when the pressure reaches the inflection point located at 4–5 MPa. It was observed that the inflection pressure of the isotherm increases rapidly as the temperature increases, while the gain in storage capacity of methane decreases. Therefore higher temperatures are not favorable for storage purpose. Hence, even though the study paves the way for a new approach in methane (co-) adsorption, there is no evidence of a practical application of these results at normal ambient conditions.

This work aim is to suggest the best adsorbent with the best efficiency and the highest desorption rate of pure methane gas for vehicular uses or in alternative for static applications. Starting from results already obtained by Inomata et al. [23] we tried to optimize the production process, varying both the starting material and the synthesis parameters.

In this way we obtained, step by step, what we consider the best results in this field up to know. In fact, as we will show later in the

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