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Artichoke (*Cynara cardunculus* L.) fibres as potential reinforcement of composite structures

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ABSTRACT

The aim of this paper is to examine the use of artichoke fibres as potential reinforcement in polymer composites. The fibres are extracted from the stem of artichoke plant, which grows in Southern Sicily. In order to use these lignocellulosic fibres as potential reinforcement in polymer composites, it is fundamental to investigate their microstructure, chemical composition and mechanical properties.

Therefore, the morphology of artichoke fibres was investigated through electron microscopy, the thermal behaviour through thermogravimetric analysis and the real density through a helium pycnometer. The chemical composition of the natural fibres in terms of cellulose, lignin, and ash contents was determinated by using standard test methods.

Finally, the mechanical characterization was carried out through single fibre tensile tests, analysing the results through statistical analysis.

1. Introduction

The use of natural fibres as reinforcement of composite materials, instead of the synthetic ones (i.e. glass, carbon or Kevlar fibres), has received growing attention in the last few years thanks to their specific properties, their price, their advantages for health and their recyclability [1]. There is a wide variety of different natural fibres which can be applied as reinforcements. The most widely used are flax, hemp, jute, kenaf and sisal, because of their properties and availability. Some recent scientific works advance the feasibility to use other natural fibres, such like okra [2] and isora [3], as reinforcement for composite materials.

The aim of this study is to identify microstructure, chemical composition and mechanical properties of a new kind of fibres, extracted from the stem of a plant of the *asteraceae* family (*Cynara cardunculus* L. var. *scolymus* L.), to make it possible to use them as potential reinforcement in polymer composites.

Globe artichoke is a plant whose capitula, commonly referred to as '*heads*' or '*buds*', are consumed worldwide as a fresh, canned or frozen vegetable. The major use of globe artichoke is for human food, but other potential uses are as a source of fresh biomass, as forage for livestock, as a feedstock for the preparation of alcoholic beverages, and as a source of inulin. Furthermore, many references exist in pharmacopoeia describing its health-promoting properties. Italy is the leading producer of globe artichoke, followed by Spain and France. In Italy (mainly Apulia, Sicily and Sardinia), artichoke production is an important activity for economic stability and social development and, thanks to its long growth cycle, its cultivation provides employment opportunities almost the whole year round [4].

These fibres have been chosen for several reasons:

1. the cultivation of artichoke is a local activity. This is important in the development of a sustainable material because allows to reduce not only the costs but also the environmental impacts (i.e. costs and impacts of transportations);
2. as shown previously, the artichoke is cultivated for many applications: mainly as food. For this, its cultivation avoids the "cash crop" (i.e. crop which is grown for profit. The term is used to differentiate from subsistence crops, which are those grown as food);
3. in 2007, the Sicily has produced about 1.5 billion of "heads". Consequently the potential market of this crop is huge (i.e. about € 173 million), due both to its great use in traditional cuisine and to its health value;
4. the fibres are extracted by the stems that represent a waste product. For the large quantities, their disposal is difficult.

The idea of the authors is to recover these stems in order to extract the fibres verifying the possibility to use them as reinforcement of composite structure.

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2. Materials and methods

Artichoke plant has been collected in a plantation in the area of Niscemi (Sicily). They belonged to the varietal type '*Violetto* di Sicilia', one of the seven Sicilian kinds of globe artichoke identified.

After collecting the fresh plant, the stem was removed and kept under water to allow microbial degradation for 25 days in order to allow fibre extraction. The stems were then washed with deionised water, dried in open air and kept in moisture-proof container (Fig. 1) to isolate the fibres (with a length between 100 mm and 160 mm).

Like the other natural fibres, those extracted from the stem of the artichoke plant have a complex structure, consisting principally of cellulose, hemicellulose, lignin, pectin and other compounds. Each fibre consists of helically wound microfibrils of cellulose, bounded together by an amorphous lignin matrix; the hemicellulose acts as a compatibilizer between cellulose w lignin [5] while pectin is also a bonding agent.

The real density of artichoke fibres was measured using gas intrusion under helium gas flow with a Pycnomatic ATC Thermo Electron Corporation equipment pycnometer. Five measurements were conducted at 20 °C.

The cellulose content of artichoke fibres was calculated by means of the density method suggested by Mwaikambo and Ansell [6]. This method allows to determine the cellulose content by measuring the real (with the technique of helium pycnometry) and apparent density (with the Archimedes method using benzene as a solvent) of the artichoke fibres.

Thermogravimetric analysis (TGA) was carried out to define the thermal stability of artichoke fibres by using a thermobalance TG/DTA Perkin Elmer 6000. Particularly, samples of weights between 2 and 5 mg were placed in a alumina pan and heated from 30 to 700 °C at a heating rate of 10 °C/min in air atmosphere.

The microstructure and morphology of the fibre were investigated by scanning electron microscopy (SEM) using a FEI QUANTA 200 F. Before analysis, each fibre was cut to a height of 10 mm, coated with gold and rubbed upon a 25 mm diameter aluminium disc.

Fourier transform infrared spectrometry (FTIR) was carried out on artichoke fibres to analyse the chemical structure of their components. IR spectrum of the fibres was recorded at the resolutions of 2 cm^{-1} using a Perkin Elmer spectrometer in the frequency range 4000–500 cm^{-1}, operating in attenuated total reflectance (ATR) mode.

Thirty fibres were mechanically tested in tension, according to ASTM standards [7], using an UTM by Zwick-Roell, equipped with a load cell of 200 N, at a constant strain rate of 1 mm/min and gage length of 10 mm. The results were analysed statistically using a commercial software (i.e. Minitab) as suggested in the literature about the mechanical tests of natural fibres.

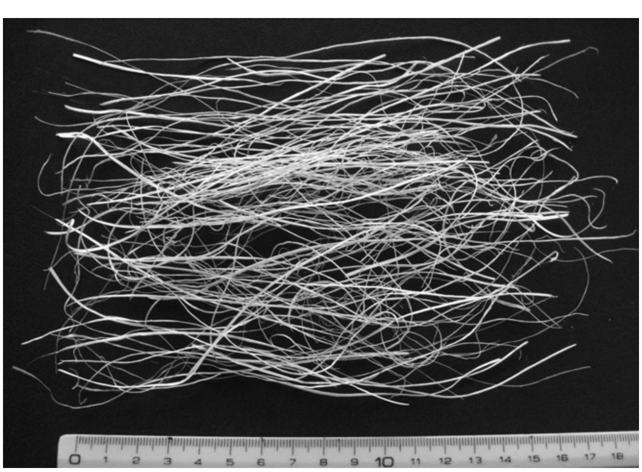

Fig. 1. Artichoke fibres isolated from the stem.

3. Results and discussion

3.1. Real density and chemical composition

The real density ρ_r of the artichoke fibres, measured using a helium pycnometer, was 1.579 ± 0.008 g/cm^3.

Benzene with a density of 0.875 g/cm^3 was used as a non polar solvent for the measurement of the bulk density of fibres and an electronic balance was used to weigh fibres. A sample of fibres was first weighted in air and then immersed in benzene solvent and reweighted.

The apparent density ρ_a of the fibres was calculated using the following equation:

$$\rho_a = \frac{\rho_s \cdot W_{fa}}{W_{fa} - W_{fs}} \quad (1)$$

where ρ_s is the density of benzene; W_{fa} is the weight of the fibres in air; W_{fb} is the weight of the fibres in benzene.

The value obtained was 1.210 g/cm^3.

The cellulose content of the artichoke fibres can be calculated using the following equation:

$$\%cell = \left[2 \cdot \left(\frac{\rho_a}{\rho_r} + \frac{\rho_r}{\rho_{cell}}\right) - \frac{\rho_a}{\rho_{cell}} - 2\right]100 \quad (2)$$

where ρ_{cell} = 1.592 g/cm^3 (density of cellulose [8]).

Substituting the known values of ρ_a, ρ_r and ρ_{cell}, the obtained value of cellulose content in the artichoke fibres is 75.62%. Obviously this one is only an indirect calculation of the cellulose content in artichoke fibres. To determinate precisely the chemical composition of the natural fibres (i.e. the cellulose, lignin, and ash contents) standard test methods were used.

In particular, the cellulose content in the artichoke fibres was determined as the Acid Detergent Fibre (ADF) according to AOAC method 973.18 [9], while lignin and ash contents were determined according to ASTM standards [10,11], respectively.

Three measures were done for determining each component and the results are reported in Table 1.

3.2. Thermal analysis

The thermal stability of artichoke fibres, one of the limiting factors in the use of natural fibres as reinforcement in composite structures [2,12], was investigated by thermogravimetric analysis. Fig. 2 shows the TG and DTG curves of artichoke fibres.

The DTG curve of artichoke fibres shows an initial peak between 40 and 100 °C (loss in weight about 5%), due to the vaporization of water in the fibre. After this peak, the curve exhibits four decomposition steps. In particular, thermal degradation of the artichoke fibres starts at 230 °C (onset degradation temperature) and the first decomposition peak occurs at about 295 °C, due to the thermal depolymerisation of hemicelluloses and pectin and the glycosidic linkages of cellulose (10% weight loss). The second peak, at about

Table 1
Chemical composition of artichoke fibres.

Component	Content (%)
Cellulose	75.3 ± 1.2
Lignin	4.3 ± 0.5
Ash	2.2 ± 0.05