

Review Article

Natural carbon fibres—An overview

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Abstract: The purpose of this review is to introduce the properties of carbon fibre, describe its application and present an ecological way of obtaining this product. This paper describes the formation of carbon fibres from various precursors. Currently, the most widely used pre-cursor is polyacrylonitrile. This is due to the fact that the obtained fibre has very good strength parameters. In opposition to the non-organic source, a renewable material is presented, which is lignin. This review highlights the advantages of using this ecological precursor and presents the general properties of the obtained carbon fibre.

Keywords: carbon fibres; manufacturing; polyacrylonitrile; lignin; biomass

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1. Introduction

The history of carbon fibre began in 1860, when Sir Joseph Wilson Swan produced carbon fibre based on cotton fibres. The process of heating the precursor occurred without the presence of air. Then, in 1879, Thomas Edison patented an incandescent light bulb with a graphite filament. The carbon fibre was made from a cellulosic precursor. The prototype required an extended lifetime, so scientists continued to research the materials. The most durable fibres turned out to be those made from Japanese bamboo. In the early 1900s, carbon fibre was replaced in the incandescent bulb by filament, so fibre was no longer such an interesting material. This lasted until 1958, when Roger Bacon grew thin carbon whiskers. He described them in his publication as rolled-up sheets of graphite. The strength of such a whisker was said to be 20 GPa, and Young's modulus was 700 GPa. It was undoubtedly a milestone invention and as a consequence very expensive. A year later, Ford and Michell patented a process in which carbon fibre was created from rayon. In contrast to the previous precursor (cotton and bamboo), rayon is a continuous fibre. Rayon fibre is a raw material for the production of carbon fibres due to its availability, low cost and non-melting nature^[1]. In 1964, Bacon and Schalamon expanded the process by heat stretching and orienting graphite layers. As a result, "Thornel 25" carbon fibre with Young's modulus of 200 GPa was introduced to the market. During this time, the US Air Force patronized the research and development of carbon fibre as a material used in composites for rockets, missiles and military equipment^[2].

2. Carbon fibres

The atomic structure of a carbon fibre depends on the material from which it is made and the manufacturing method. It is similar to the structure of graphite, but it is less ordered. Carbon fibre structure consists of the basic structural units of carbon planes. The distance between the turbostratic planes is typically >0.34 nm, while the distance between perfect graphite planes is 0.3345 nm^[3].

Various substrates are used to produce such a fibre. These can include polyacrylonitrile (PAN), mesophase pitch, synthetic polymers like polystyrene, polyethylene, and polyacetylene. Eco-friendly materials are also being used. One of them is lignin. It is further described in this paper. The variation in the properties of the precursor causes a change in the properties of the obtained fibre. It is therefore obvious that the properties of a fibre made of polyacrylonitrile will not be exactly the same as those made of cellulose. Nevertheless, the general basic parameters are similar. To optimize the creation process, modifications are also created. For example, lignin is blended with another polymer to increase the average tensile strength of the finished fibre. **Figure 1** shows the most commonly used precursors for carbon fibre with the obtained average tensile strength^[4].

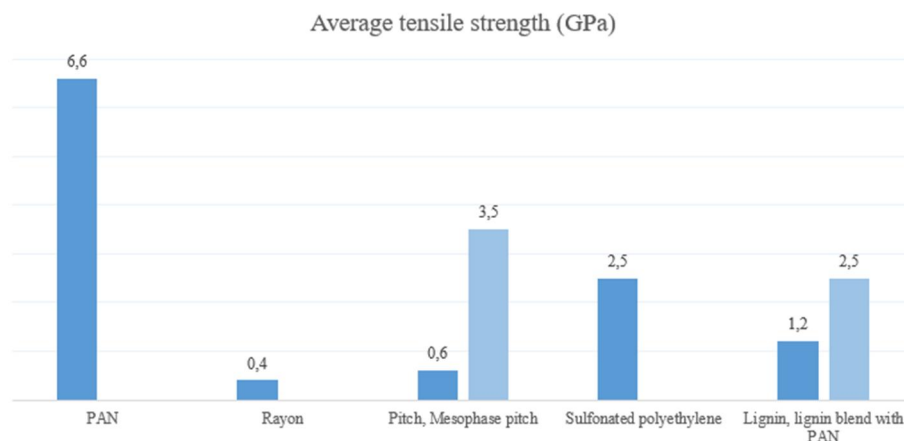


Figure 1. Comparison of different carbon fibre precursors and achieved average strength^[4].

Depending on the carbon content, two groups are distinguished: carbon and graphite. The first of these contains 80%–98% of carbon, and graphite fibres contain about 99% of this element. The high carbon content in the composition ensures that the fibres are infusible and chemically resistant.

Carbon fibres are characterized by high tensile strength, high Young’s modulus, high fatigue strength, and high creep strength. These parameters depend on the degree of graphitization. The processes of carbon fibre formation will be described in the paper. It is possible to obtain a carbon fibre with a high Young’s modulus value but lower tensile strength or the contrary. Fibres with ultra-high modulus are obtained as a result of high graphitization. They are then called graphite fibres. Carbon fibres are classified among those with high modulus.

They have good heat and chemical resistance. Carbon fibres have a low density, and low dead weight. Further advantages are the ability to dampen vibrations and the fact that they do not absorb (to a large extent) X-rays.

Carbon fibres can be divided according to their final mechanical properties^[5]:

- UHM, ultra high modulus >500 GPa;
- HM, high modulus >300 GPa;
- IM, intermediate modulus >200 GPa;
- LM, low modulus >100 GPa;
- HS, high strength >4 GPa.

There is also a division according to the final heat treatment temperatures:

- Type I—heat treatment at 2000 °C;

- Type II—heat treatment at 1500 °C;
- Type III—heat treatment at 1000 °C.

Type II PAN carbon fibres are typically high strength carbon fibres while most high modulus carbon fibres are type I.

3. Conventional carbon fibre manufacturing

3.1. Polyacrylonitrile

Producing carbon fibre with the desired properties involves several steps. The first is the selection of a precursor. Primarily, polyacrylonitrile (PAN) is used to produce carbon fibre. Its properties, high carbon yield and ability to decompose to form char prior before melting indicate that polyacrylonitrile is the precursor from which the carbon fibre will be obtained will have the best properties^[4]. It accounts for about 90% of worldwide production^[6].

Polyacrylonitrile is a polymer obtained by radical polymerization of acrylonitrile. The most common in application is polyacrylonitrile containing 68% carbon^[7]. PAN can be produced by polymerization in solution, suspension and/or emulsion^[2]. It can be polymerized from acrylonitrile using initiators such as peroxides and azo compounds, through the process of addition polymerization^[7]. Carbon fibres made from a precursor polymer owe their properties to their molecular structure containing highly polar nitrile groups. They cause strong interactions that maintain the orientation of the polymer chains at high temperatures. PAN should have a high molecular weight of more than 200 kDa. This results in high crystallinity and high strength. However, they are difficult to dissolve due to their high polydispersity, so one with a molecular weight of less than 200 kDa is also used^[4].

At an ambient temperature of 220–250 °C, the product is heated for about 50 h. This leads to heat stabilization and complete oxidation. The stabilization process is an exothermic process, so the heating procedure must be under control. The use of a high heating rate generates a large amount of heat in a short time. This can lead to a decrease in carbon yield and bring defects into the precursor fibres. Therefore, a lower heating rate is preferable. It will allow oxygen to diffuse into the core of the precursor fibres to complete stabilization^[7].

Next, the carbonization process begins, leading to the removal of non-carbon components. The inert ambient temperature is then increased to more than 1000 °C. The strength of the fibre will increase with the carbonization temperature. At a temperature of 1500 °C, maximum strength is observed. At higher temperatures, the tensile strength will decrease and the modulus will have a higher value. The time at which the fibre will be heated depends on the diameter of the fibre and its composition. A process that is too fast can lead to the destruction of the precursor, while a slow carbonization process results in the loss of too much nitrogen in the early stages of carbonization, a certain amount of which is preferred to obtain high-strength carbon fibres. The mentioned fibre diameter will gradually decrease as a result of the removal of non-carbon elements^[7].

Then comes the graphitization process. It leads to obtaining a higher modulus. It involves heating the fibres to temperatures ranging from 2000 °C to as high as 3000 °C. By achieving the appropriate degree of graphitization, the material obtains the intended properties, structure, an increased crystalline orientation in the fibre direction, and a reduction in interlayer spacing and void content^[7].

When the fibres are carbonized, they have a surface that does not bond well with epoxies and other materials used in composite materials. To allow the fibres to bond with other materials, their surfaces are

treated (oxidized). Oxidation can be achieved by immersing the fibres in various gases or liquids. These can involve air, carbon dioxide or ozone, sodium hypochlorite or nitric acid. The surface treatment process, like the whole process of creation, must be carefully controlled^[8].

Polyacrylonitrile is undoubtedly the most important carbon fibre precursor at this point. This is due to its high carbon content, which has begun to be used commercially. However, the production of carbon fibre from polyacrylonitrile involves rather high production costs. It also has the disadvantage of requiring a harmful solvent and a long graphitization time^[9].

3.2. Mesophase pitch

Polyacrylonitrile, however, is not the only known precursor. For the production of carbon fibre, properly refined mesophase pitch is used as a substrate. Natural pitch is a by-product of the coal and oil industries, while synthetic pitch is manufactured through the pyrolysis of synthetic polymers. The application of synthetic pitch has recently attracted more interest because of its higher purity. In addition, the stabilization process can proceed at a faster rate at a given temperature^[7]. As a substrate, it provides the receipt of carbon fibres with a sheet structure, high modulus and low strength. Only transformed mesophase pitch ensures their high strength. Pitch accounts for about 10% of carbon fibre production. It is used mainly because of its high carbon yield. Together with polyacrylonitrile, it accounts for most of the world's carbon fibre production^[4]. Carbon fibre is formed from mesophase pitch in a similar way to that formed from polyacrylonitrile. It is obtained by heat treatment, followed by carbonization and graphitization. The process of creating such a fibre is shown in **Figure 2**^[10].

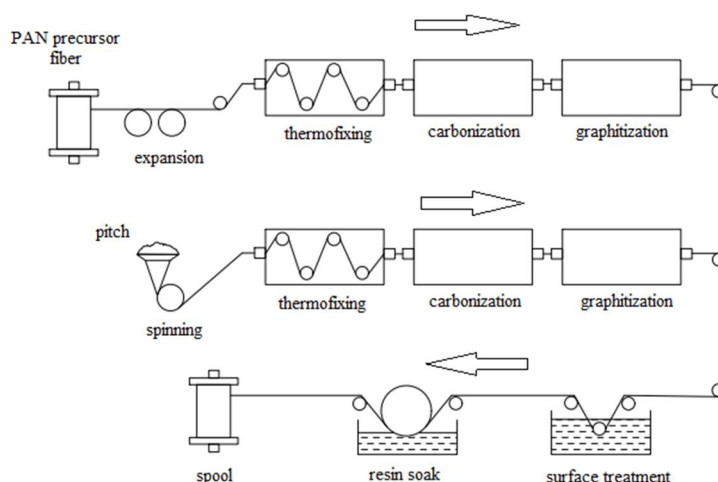


Figure 2. Scheme of the process of obtaining carbon fibres from polyacrylonitrile PAN and pitch, basing on the study of Dobrzański^[10].

4. Carbon fibre formation from lignin

Using polyacrylonitrile to manufacture carbon fibre has its benefits and drawbacks. The cost of producing a carbon fibre is half the price of the precursor^[11]. Efforts are being made to find the most cost-effective material that is also a renewable source. Lignin is one of the most popular eco-friendly precursors for producing carbon fibre. Cellulose fibres leave a smaller carbon footprint compared to polyacrylonitrile fibres^[12].

Lignin is a renewable, plant-based material, whose annual worldwide production is about 600 billion tons, where the use in the paper industry is around 70 million tonnes^[13,14]. The first carbon fibre based on lignin was

created in 1964, in Japan. In the 1970s, Nippon Chemical Co. created the first commercially produced lignin-based carbon fibre^[15]. Currently, the vast majority of lignin is used as cheap fuel for combustion^[14].

Lignin, cellulose and hemicellulose are the three main components of the plant cell wall. Lignin occurs in the form of a black, very thick liquid. It takes this form by occurring as a by-product of the hydrolyzed wood and paper industries. In plant cells, it is white or almost clear. There it has the functions of a filler and a binding agent. Its purpose is to strengthen the cells and stem of the plant, giving them high mechanical strength. In addition, it also protects against biological and chemical damage^[14]. The material is extracted from wood, making it an environmentally friendly material from a renewable source. The type of wood influences the lignin content. This ranges from about 10% to 40%. Lignin content is higher in coniferous trees than in deciduous trees. For example, the lignin content of spruce wood states 29%–34% of dry material^[16], while for birch wood, it is 24%–26%^[17]. The high carbon content of kraft lignin (about 60%–65%) suggests a high yield when converted into carbon fibre, while the properties of the resulting fibre are not as strong as those formed from PAN^[18].

Not only the lignin content is variable, but also its composition. Two main types of subunits can be distinguished in lignin. One is derived from coniferyl alcohol monomers, forming guaiacyl (G) units resulting in the lignin typical of coniferous wood. The other subunit, the sinapyl alcohol monomer, forms syringyl (S) units after polymerisation. The hybrid type of lignin with units of G and S is typical of hardwoods and is distinguishable by the ratio of the subunits (S/G ratio)^[19].

4.1. Lignin extraction

Extracting lignin from plants can be done in two ways. The first involves dissolving and removing components other than lignin. This yields hydrolyzed lignin in the form of an insoluble component. The second method is to dissolve the lignin among other insoluble components^[14]. By the first method described above, mostly kraft lignin is obtained. The second method yields alkaline lignin, lignin sulfonate, organosolv lignin, and steam explosion lignin. **Table 1** shows the characteristics of the various types of lignins^[14].

Table 1. Types and features of lignin^[14].

Lignin type	Characteristics
Alkali lignin	It is characterized by many hydroxyl groups, strong hydrogen bonds, and the water solubility depends on the molecular weight.
Organosolv lignin	It is characterized by high purity, high solvent cost, and complicated recovery.
Kraft lignin	It is characterized by high phenolic hydroxyl content, low methoxyl and alcoholic hydroxyl content and high purity.
Lignin sulfonate	It is characterized by the presence of an amphiphilic surfactant polymer molecule. Its molecular structure is severely damaged during pulping.
Steam explosion lignin	It is characterized by high requirements on equipment and energy consumption.

As a result, the obtained fibre is not used in industries where high material performance is necessary. Carbon fibres formed from a renewable precursor will have applications wherever low density and medium mechanical properties are required.

4.2. Conversion

The conversion of lignin into fibre can begin with three methods: melt spinning, solution spinning and electrospinning.

4.2.1. Melt spinning

The most popular method is melt spinning. This is a process in which material consumption is high, with low air pollution^[20]. Manufacturing by melt spinning often requires fractionation, chemical modification or blending with polymers/additives for successful melt spinning^[18]. However, it has its disadvantages. Additives can be added to improve spinning but only in small amounts. Otherwise, they can interfere with the carbonization process. The purer the lignin is, the longer the stabilization process is^[2]. About 100 h must be spent on the stabilization step to achieve relatively high quality^[18].

4.2.2. Solution spinning

Solution spinning can be divided into wet spinning and dry spinning. Using wet spinning, the fibre diameter is controlled and a small amount of solvent remains in the fibres. Dry spinning leaves more voids and more solvent in the fibres. This method is used using lignin with higher mechanical properties^[21]. In general, the speed of spinning for the dry-wet method is 2–10 times higher than for the wet method. This is beneficial from an economic point of view—for reducing production costs^[13]. The main thing is that the properties and morphology of solution-spun lignin fibres are affected by two aspects. The first major factor is the internal structure (that is, the chemical structure of the lignin, molecular weight, etc.), which depends on the lignin type and the methods of extraction. The second is the processing parameters (content, temperature, condensation and stretching)^[22].

4.2.3. Electrospinning

The third method is electrospinning. It is used to produce fibres with very small diameters. It consists of spraying and spinning the material in a strong electric field. Then the drop of solution at the tip of the needle turns into the so-called “Taylor’s cone^[14]”. Lignin fibres are produced once the solvent in the solution has volatilized during spraying^[22]. Lignin fibres can also be prepared using other methods, e.g., melt-blow, centrifugal spinning, barrel spinning, and phase inversion extrusion^[13].

4.3. Pre-oxidation

Next comes the longest and very important process of pre-oxidation. During this time, the fibre structure is stabilized and its mechanical properties are improved. Proper selection of temperature improves its mechanical properties. It is usually around 200 °C to 300 °C. This is an essential step that ensures that the material will not melt during carbonization. Then the lignin becomes thermally rigid. As with the production of the fibre from PAN, it should be a good practice for the stabilization step to be slow, so that it does not cause the degradation of the fibre’s quality^[9]. The reaction occurring during thermostabilization for lignin differs from that for the precursor pitch. In the case of the first one, there is a partial loss of mass during the entire stabilization. In the case of pitch use, there is an increase in quality, since alkyl oxidation occurs before the processes of dehydrogenation and cyclization^[13].

4.4. Carbonization

During the next stage, which is carbonization, the material is treated. The purpose of this treatment is to obtain a disordered graphite structure^[23]. Carbonization is divided into two steps. The first is low-temperature carbonization (300 °C–1000 °C), and the second is high-temperature carbonization (1100 °C–1600 °C). Carbonization and graphitization are similar stages. Depending on the temperature, they have different degrees

of orientation and crystallization. Carbonization enriches the percentage of carbon, while graphitization transforms this carbon into graphite. The duration of the process should be optimized enough to obtain high modulus and strength.

4.5. Surface treatment

The next step is surface treatment. It is performed to achieve a good interaction between the fibre and the polymer matrix. This can be done by oxidative or non-oxidative methods. After processing, the fibres are covered with a thin layer of resin. These processes are designed to improve the mechanical properties of the fibre, ensure proper interaction between the fibre and the strengthened composite, and to make it easier to process^[9].

4.6. Sizing

The final stage in the production of carbon fibres involves protecting the fibres from damage during winding or weaving. The protective materials must be compatible with the adhesive used in creating the composite. Such materials often become epoxy, polyester and nylon^[8].

5. Other ecological precursors

5.1. Rayon

Cellulosic fibres such as cotton, flax and sisal are also used as precursors. The most extensively studied material of these was rayon. Artificial silk is made from the cheapest cellulose, wood pulp, by solution spinning^[7]. The transformation of rayon into carbon fibre is similar to that from polyacrylonitrile. It involves thermal decomposition/oxidation, carbonization and graphitization. However, the whole process is very expensive due to the low yield of the resulting carbon fibres. Therefore, rayon is often chemically modified to increase the strength of the obtained product by up to 28%^[4]. The manufacture of carbon fibre based on rayon is a very expensive process. This is due to the expensive tensile high-temperature processing. Carbon fibre based on this precursor can achieve an elastic modulus of more than 500 GPa with an average tensile strength of 2.5 GPa^[7].

5.2. Viscose

Viscose-based fibres were first developed in the mid-20th century. As a precursor, viscose is still widely known and used today. It is formed from wood pulp and cotton. Viscose-based carbon fibres are transformed by processes such as polyacrylonitrile. These stages are joined by two additional ones: washing and impregnating the catalyst^[24].

5.3. Linen

Linen fibres are used to create lightweight structures. They have very good damping properties and specific mechanical properties similar to polymers strengthened with synthetic fibres. The following properties are due to the low specific weight of flax fibres and their highly dissipative nature. Prefabricated linen textiles are soaked in resins using various methods. Composites reinforced with flax based fibres show anisotropic properties, which is why they are often combined into woven textiles forming transverse and multidirectional laminates^[25].

5.4. Sisal

The other renewable precursor is sisal. Sisal is a very strong fibre that comes from a plant called sisal agave. A chemical reaction with zinc chloride produces activated carbon with a wide surface area and numerous pores^[15].

5.5. Jute

For the production of carbon fibres based on natural fibres, the jute is being used. It is a material containing mainly lignin and cellulose, which are already widely used for this purpose. Jute fibre is one of the cheaper bio-based fibres. In comparison with other natural precursors, it has a high Young's modulus^[15].

5.6. Coconut shell

An unconventional precursor to carbon fibre has turned out to be coconut fibre. It is a fibrous substance that is taken from the shell of this nut. Most of this raw material is burned as fuel. Processing coconut fibre may produce a high-performance carbon fibre with good thermal stability and less shrinkage after treatment. Not only the coconut shell can be a precursor but also the peanut shell. To form carbon fibre, the nuts must first be powdered and then subjected to chemical reactions^[15].

5.7. Bagasse

The next one is a bagasse. They are fibrous leftovers from the extraction of cane juice. In their composition are lignin, cellulose and hemicellulose. To produce carbon fibre from such a precursor, the lignin that goes into it must first be extracted from it. It is then processed as described in the previous section of the paper^[26].

5.8. Banana peels

Moreover, the composition of banana peels allows them to be used as activated carbon—they contain about 41% of this element. The banana stalk consists of cellulose, hemicellulose and lignin. Research has also been conducted on this material as a potential precursor for carbon fibres^[15].

6. Application

In the 20th century, the main use of carbon fibre composites was in military centres and NASA. Currently, the discussed fibres are used in many areas of daily living, for example in medicine^[27], civil engineering^[28], sport^[29]. Carbon fibres are widely used in aviation^[30], in the production of yacht components (rudders, masts). In sports, carbon fibre reinforced composites are used to make equipment, for example, bicycle parts, surfboards, golf clubs, fishing rods, and sports shoes. They are also used in medicine. Due to the lightness and strength of the material, they are ideal for the production of prostheses and orthopedic splints^[31]. Its properties and appearance are appreciated by automotive fans^[32]. In this field, the use of carbon fibres to equip Formula 1 vehicles is widely known. Also, their strength and lightness are balanced in electric vehicles, where batteries overwhelm their weight^[2]. A number of properties of carbon fibres allow the use of this material in construction. These are relatively new solutions. Fibres may be a replacement for traditional steel reinforcement in the form of bars or meshes^[33]. They are used in strengthening and reinforcing structures. Especially in bridge structures, the advantages of carbon fibre can be used because in these constructions corrosion resistance is very important^[34].

7. Conclusion

Carbon fibres find their application in many areas of life. They are used by strengthening other materials to form high quality composites. Properly prepared carbon fibre is characterized by high strength, modulus, low density, high temperature resistance, corrosion resistance, good thermal and electrical conductivity, vibration damping ability and low X-ray absorption. Their properties are affected by how they were formed and from what precursor.

There are currently two main precursors to carbon fibre production. The first, most commonly used, is polyacrylonitrile (PAN), and the mesophase pitch (MP) is the second. This is due to the fact that the use of these materials gives the most satisfactory parameters of the obtained fibre. Unfortunately, the use of PAN and MP has its drawbacks, due to which scientists are looking for other solutions. Crude oil is an expensive precursor material because its resources are not renewable. The cost of producing a carbon fibre is half the price of the precursor. Therefore, using a renewable, cheaper material is key here. Currently, there is a growing interest in cheap, ecological substitutes for polyacrylonitrile. Cellulose fibres are cheaper and leave a smaller carbon footprint compared to polyacrylonitrile fibres. Nowadays, consumers are paying attention to living in harmony with the environment. Therefore, interest in carbon fibres based on precursors produced from biomass is certain to increase. The most common source of biomass is lignocellulosic biomass, which consists of wood, agricultural and forestry waste. Most biomass materials contain a mixture of cellulose and lignin derivatives. They are very attractive due to their high carbon content and low price.

The best known and studied precursor is lignin. It seems to be a very good substitute for the above-mentioned materials. It can be obtained by various means from many precursors. Much research is needed to optimize the process of carbon fibre formation. In order to obtain a higher quality carbon fibre, it is necessary to ensure that the quality of the precursor fibre is high enough. To reduce costs and minimize use, the spinning process should be properly controlled. This can affect the properties of the fibre without the need for harsh surface treatment, high temperatures during further processing of the fibre, or the use of advanced chemicals.

Research has been done on other natural fibres as precursors to carbon fibre. These included silk, chitosan or eucalyptus tar. However, these environmentally friendly and renewable materials did not yield properties like polyacrylonitrile. It is very important to look for new solutions based on biomaterials. By reducing the use of petroleum, which is an expensive material, they reduce carbon emissions. Cellulose fibres leave a smaller carbon footprint compared to polyacrylonitrile fibres. Therefore, the use of a green precursor is good for the environment and economical. The cost of producing a carbon fibre is half the price of the precursor. Nevertheless, a lot of research still needs to be done to optimize the production processes in order to obtain a product with satisfactory parameters in the least amount of time, at the lowest cost, and using the maximum potential of the precursor.

Conflict of interest

The authors declare no conflict of interest.

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