METHODS FOR SYNTHESIZING GRAPHENE FROM A LIGNIN SOURCE

RELATED APPLICATION

[0001] This application claims priority from U.S. Provisional Patent Application No. 61/831,297, filed Jun. 5, 2013, the entire disclosure of which is incorporated herein by this reference.

STATEMENT OF GOVERNMENT SUPPORT

[0002] The invention described herein was made with government support under grant Nos. 114V-11111124-097 and 124V-11111124-091 awarded by the USDA Forest Service. The government has certain rights in the invention.

TECHNICAL FIELD

[0003] The presently-disclosed subject matter relates to methods for synthesizing carbon nanomaterials from wood by-products. In particular, the presently-disclosed subject matter relates to the use of lignin from pulping operations for synthesizing carbon-based nanomaterials, such as graphene nanosheets.

BACKGROUND

[0004] Lignin is a renewable carbon source and the second most abundant biopolymer next to cellulose on earth. It is a crosslinked and complex natural polymer composed of a phenyl propane unit as a basic unit, and lignin includes about 65 percent carbon, 6 percent hydrogen, and 29 percent oxygen. A great amount of lignin can be obtained from the pulping industry in the name of kraft lignin (or thiolignin) and lignosulfonate (LS). The approximate amount of lignin production in the existing pulping industry worldwide is estimated at more than 50 million tons/year. However, the complex structure of lignin makes the production of value-added chemicals from lignin difficult. Therefore, most lignin is currently not isolated, but is instead burned onsite to recover pulping chemicals and provide steam for power production in kraft pulp mills.

[0005] In this respect, LS from the sulfite pulping process is a largely available source of lignin, and the global production of LS is currently around 1 million tons/year. LS has been used for concrete admixtures, dispersants and dust suppression for roads, pellet binders, and vanillin. Others have also attempted to convert lignin into value-added materials, but there are few reports on the successful commercialization of lignin-related products. In particular, there are currently no systematic approaches in the processing of lignin for conversion into carbon-based nanomaterials, including the conversion of lignin to graphene. Additionally, carbon nanomaterials, such as graphene, have stimulated considerable scientific, industrial, and commercial interest due to their intriguing physical properties and enormous potential for various applications. Therefore, it would be beneficial if lignin could be utilized as an alternative carbon source for the production of high value carbon-based nanomaterials and the like.

[0006] Accordingly, there remains a need for systems and methods that can convert lignins and/or sources thereof to carbon nanomaterials, such as graphene. It would be advantageous if systems and methods could be achieved to convert lignins to carbon nanomaterials using simple and cost effective solutions.

SUMMARY

[0007] This summary describes several embodiments of the presently-disclosed subject matter, and in many cases lists variations and permutations of these embodiments. This summary is merely exemplary of the numerous and varied embodiments. Mention of one or more representative features of a given embodiment is likewise exemplary. Such an embodiment can typically exist with or without the feature(s) mentioned; likewise, those features can be applied to other embodiments of the presently-disclosed subject matter, whether listed in this summary or not. To avoid excessive repetition, this summary does not list or suggest all possible combinations of features.

[0008] The presently-disclosed subject matter provides, in some embodiments a method of synthesizing carbon nanomaterials, the method comprising mixing a lignin and/or source thereof and a catalyst to form a mixture heating the mixture at a temperature of at least 600° C. for at least 30 minutes, and cooling the heated mixture to form a cooled mixture including graphene. In some instances the lignin and/or source thereof can be selected from a kraft lignin, a thiolignin, a lignosulfonate, a sulfur-free lignin, and combinations thereof, including sodium lignosulfonate.

[0009] Additionally, in some embodiments the catalyst can selected from a platinum-containing catalyst, a nickel-containing catalyst, an iron-containing catalyst, and combinations thereof. The catalyst can also be comprised of nanoparticles. For example, the catalyst can include iron nanoparticles, iron nitrate, or a combination thereof. In certain embodiment s the lignin and/or source thereof and catalyst are in a weight ratio of from about 1:1 to about 8:1, such as a weight ratio of about 4:1. In certain embodiments the heating is conducted at a temperature of from about 600° C. to about 1500° C., such as at a temperature of about 1000° C.

[0010] In some embodiments the heating is conducted under an inert atmosphere. In some embodiments the heating is conducted for a period of from about 30 minutes to about 120 minutes. In some embodiments the cooling is conducted under an inert atmosphere. In some embodiments, in the cooling step, the mixture is cooled to a temperature of less than about 100° C.

[0011] Embodiments of the presently-disclosed methods can further comprise, after the cooling step, a step of purifying the mixture to increase a concentration of carbon nanomaterials in the cooled mixture. For example, the purifying step can include purifying the mixture with water, an acid, or a combination thereof.

[0012] Other embodiments of the presently-disclosed subject matter provide a method of synthesizing carbon nanomaterials, the method comprising mixing sodium lignosulfonate and iron nanoparticles to form a mixture heating the mixture at a temperature of about 80° C. to about 1200° C. for about 40 minutes to about 80 minutes under an inert atmosphere and cooling the heated mixture to a temperature of less than about 100° C. under an inert atmosphere to form a cooled mixture including graphene. In some instances the iron nanoparticles have a diameter of less than about 100 nanometers.

[0013] Other embodiments of the presently-disclosed subject matter provide a method of synthesizing carbon nanomaterials, the method comprising mixing kraft lignin and an iron-containing catalyst to form a mixture, heating the mixture at a temperature of about 80° C. to about 1200° C. for about 40 minutes to about 80 minutes under an inert atmo-