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Review

Review of Carbon Nanotube Research and Development: Materials and Emerging Applications

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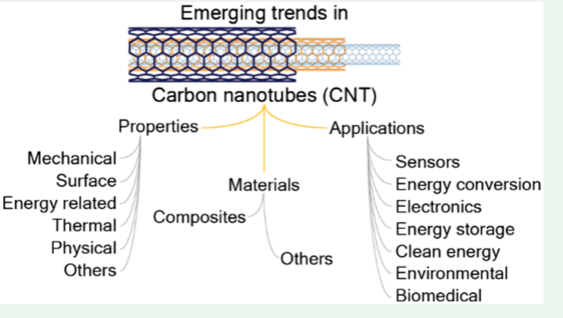
ABSTRACT: In this study, we present our findings from analyzing data contained in approximately 265,000 journal and patent publications in the field of carbon nanotube (CNT)-related research spanning the last two decades (2003 to 2023). The purpose of this study is to identify and extract prominent trends and establish connections, such as those between materials and applications. Using a natural language processing (NLP)-based analysis, we have identified over 80 emerging concepts across three major categories (applications, materials, and properties) in the field of CNTs, which is presented in a "Trend Landscape" map. While early research efforts appeared to focus on synthesis techniques (including the now well-established and dominant technique of chemical vapor deposition (CVD)), in recent years the field has been driven by an increase in interest in CNT composites, especially involving other nanoscale materials such as MXene and metal−organic frameworks (MOFs). High growth applications include the use of CNTs in energy storage and conversion technologies such as fuel cells, next-generation batteries, and nanogenerators, and in sensors including wearable human motion sensors. Investment data suggests commercial interest in development and use of CNTs in a variety of industry types, such as semiconductors, energy, and to a smaller extent the biomedical industry. The intent of this report is to serve as a useful guide to researchers to aid in further exploration of the field of CNT based materials and applications.



KEYWORDS: *carbon nanotubes, SWCNT, MWCNT, carbon allotropes, 1-dimensional materials, CNT/polymer composites*

■ INTRODUCTION

Carbon nanotubes (CNTs) are a one-dimensional (1-D) allotrope of carbon, made up of a sp^2 hybridized carbon lattice in the form of a cylinder. A single-wall CNT (SWCNT), the simplest version of a CNT, consists of a single, cylindrical graphene tube. In double-wall CNTs (DWCNTs) and multiwall CNTs (MWCNTs), two or more tubes are either nested concentrically or wrapped like a scroll. SWCNTs are typically 0.4−2 nm in diameter, while MWCNTs can be much larger, with diameters of tens of nanometers, having both ends generally capped by fullerene-like domes.[1]

Similar to fullerenes and graphene, which are the analogous 0- and 2-D allotropes of carbon, the graphite-like arrangement of carbon atoms in CNTs and their shape give them remarkable and unique properties, which has resulted in the widespread use of CNTs in several fields of science and technology, including energy storage, electronics, structural composites, biomaterials, and others.

Because of their 1-D structure, CNTs have highly anisotropic mechanical, electrical, and thermal properties, which make them ideal for use in polymer, ceramic, and metal matrix composites. Additionally, their electrical properties, very high surface to volume ratio, and potential for chemical functionalization has led to their use in battery and supercapacitor components.[2,3]

In the following sections, we will discuss the trends in CNT-related research from 2003 to 2023, with a focus on identifying the emerging topics of research in this area over the last 3 years. This analysis was performed by first identifying journal and patent publications in the CAS Content Collection related to CNTs, which resulted in a set of roughly 265,000 documents. We identified emerging applications, materials, applications, properties, and other concepts in CNT-related research using a Natural Language Processing (NLP) based approach.[4,5] This analysis identified over 39,000 concepts that appear in at least 20 documents, which were then classified, counted, grouped, and analyzed. The content of the following sections is based on the results of this analysis, in combination with substances, concepts, and other information indexed in

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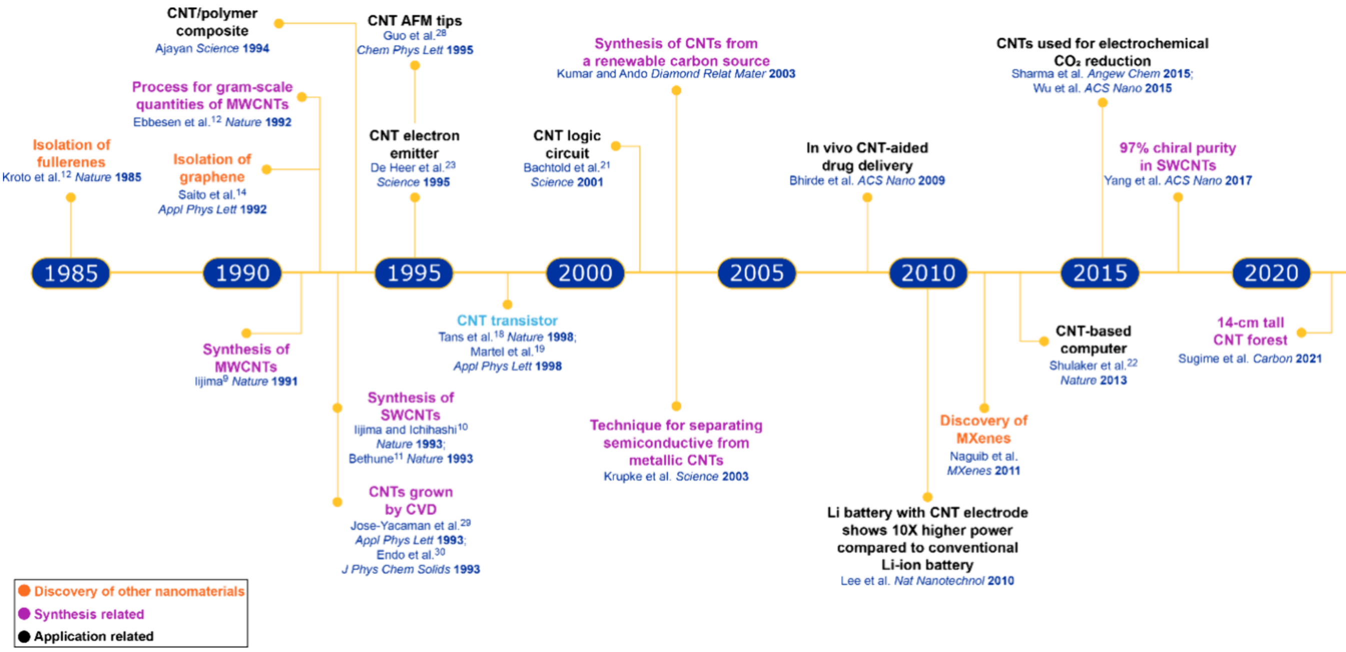

Figure 1. Timeline showing notable events in the recent history of CNTs and other notable nanoscale materials. Events have been color coded to indicate area of work (synthesis (purple), application (black) or discovery (orange)).

the CAS Content Collection. For a more detailed description of this process including specifics of the NLP methodology, please see the Methods section in the Supporting Information. This NLP methodology has been used successfully by us to identify emerging concepts in several other scientific areas including immuno-oncology,[5] biomaterials,[6] and few more unpublished materials currently in the works.

The purpose of this review is to use data extracted from the large number of publications on CNT research, data including the frequency, time evolution, and co-occurrence of key concepts identified by NLP analysis, to summarize trends and emerging connections in this field. This involves both looking back on the past evolution of major trends in CNT research and development, and to determine which topics are driving publications currently. Where possible, examples of publications that represent key trends are also provided, which were selected to highlight highly cited publications and the work of prominent research groups. Special emphasis is also placed on concepts which have seen high growth in publications in recent years. It is important to note that the perspective of this review is based chiefly on data science, rather than from experts in the field of CNTs.

The applications driving recent growth in CNT publications include energy storage, energy conversion, environmental remediation, CO_2 reduction, and sensors. Battery applications were determined to have the comparatively highest level of commercial activity, based on analysis of journal versus patent publications. Furthermore, the growth of publications related to the synthesis of CNTs appeared to slow around 2006, with a shift to more focus on solving challenges such as the optimization of performance of CNTs for specific applications, the use of CNTs in emerging applications, and their combination with other nanoscale materials.

HISTORY AND GENERAL TRENDS IN CNT PUBLICATIONS

The current era of CNT research began in 1991 when Sumio Iijima of Nippon Electrical Corporation's (NEC) Fundamental Research Laboratory in Tsukuba, Japan, synthesized MWCNTs using arc-discharge evaporation from a carbon anode and characterized them with transmission electron microscopy.[7] Subsequent research by groups at NEC and International Business Machines Corporation (IBM) in 1993 produced SWCNTs[8,9] by incorporating a metal catalyst into the cathode. Chronologically, CNTs were discovered between the isolation of fullerenes in 1985 and graphene in 2004.[10,11]

A timeline of the key events in the history of CNT research is shown in Figure 1. These include efforts to synthesize CNTs using new methods or into alternate form factors, understand the properties of CNTs through characterization and modeling, and evaluate and optimize their performance in various applications.

Shortly after their discovery, three theoretical studies provided valuable insights on the electronic properties of SWCNTs.[12−14] By calculating the electronic structures of SWCNTs, these studies predicted that individual tubes could have either metallic or semiconducting electrical properties, depending on their chirality and diameter. Furthermore, they determined that Peierls distortion, which can interfere with the electrical conductivity of 1-D metals, was not a significant effect in metal-like SWCNTs at ambient temperature. The theoretical predictions in these publications would later be confirmed by direct measurement of diameter and chirality using scanning tunneling microscopy, combined with measurement of the electrical properties of individual CNTs.[15]

Building on this work, in 1998 research groups at Delft University of Technology and IBM fabricated CNT-based field-effect transistors (FETs),[16,17] placing individual SWNTs over a silicon-based gate structure between metallic electrodes, similar to a doped Si-based FET. Over the next 15 years,[18] full

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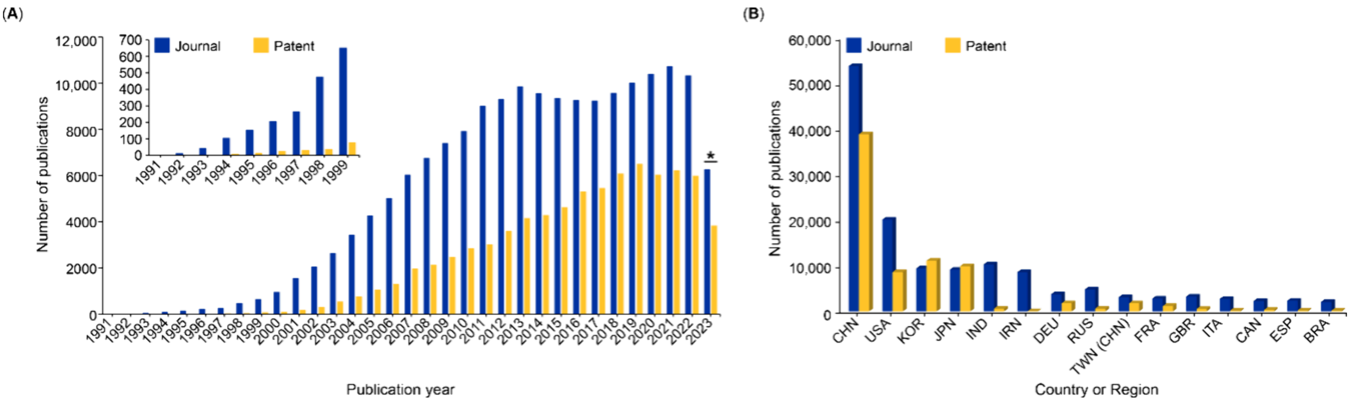

Figure 2. (A) Overall publication trend for CNT-related research between 1991 and 2023. (B) Leading countries or regions in CNT-related research between 2003 and 2023. Data includes journal and patent publications from the CAS Content Collection. **The data for 2023 is partial and only includes data from January to July.*

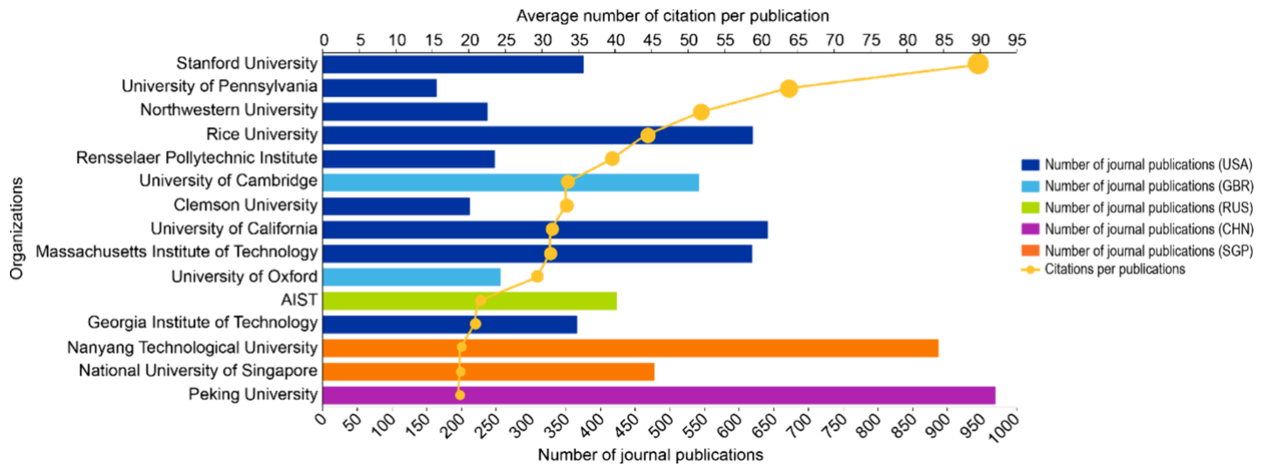

Figure 3. Research organizations leading in CNT-related research in terms of average number of citations per publication. Data includes journal from the CAS Content Collection for the period 2003 to 2023.

logic circuits[19] and eventually a computer[20] were built using CNTs as the active components.

During this time, researchers began using CNTs in other applications that benefit from their 1-D geometry, for example, using CNTs as field-emission electron sources.[21] In 1998, CNT-tipped atomic force microscopy (AFM) probes were reported, demonstrating the potential of CNTs in enhancing scanning probe microscopy capabilities owing to their inherent high-aspect-ratio geometry.[22] An innovative example of this is the use of carboxyl groups present on partially oxidized ends of the CNT AFM tips as attachment points for other chemical moieties, thereby forming AFM probes that are sensitive to the chemical functionality present on a sample surface.[23,24]

The ability to explore the use of CNTs in a wide variety of applications was enabled by advances in CNT synthesis in the 10 years following their discovery. In 1992, by optimizing the conditions of the arc-discharge process, particularly the helium pressure, Ebbesen and Ajayan were able to generate and isolate gram-scale quantities of MWCNTs.[25] Subsequently, a group at Rice University led by Richard Smalley developed a process based on laser ablation of metal/graphite composite targets to produce SWCNTs with high yield.[26]

Another major development around this time was the development of chemical vapor deposition (CVD) methods to synthesize CNTs.[27,28] In CVD, a carbon-containing precursor gas (benzene and acetylene in these early studies) flows over a substrate at elevated temperatures. Reactions between the precursor and surface then lead to the growth of CNTs from the surface. The degree of morphological control, adaptability, and scalability of CVD has led to it being the most common synthesis route for CNTs today. An example of the control and scalability of CNT CVD is the ability to generate aligned "forests" of CNTs on a substrate consisting of catalytic iron nanoparticles embedded in silica.[29] Smalley is perhaps better known for his work on developing high pressure carbon monoxide (HiPco) method of preparing SWCNTs, a type of CVD allowing large scale (10g/day) production of SWCNTs.[30]

A total count of the CNT-related journal and patent publications between 1992 and 2023 is shown in Figure 2. Research in this field grew rapidly between 1991 and 1999, to roughly 1,000 publications per year by 2000 (inset Figure 2A). Between 2000 and 2013, an approximately linear, 10-fold increase in the number of journal publications is observed. Between 2013 and 2017, growth in journal publications appear to have plateaued and remain more or less constant at 10,000 publications per year, while the frequency of patent publications continued to grow, eventually leveling off at ~7,000 per year in 2018 (Figure 2A). Since 2016, the number of publications generally increased again, driven by diverse, emerging applications including electrochemical CO_2 reduction, flexible sensors, and others that will be discussed in more

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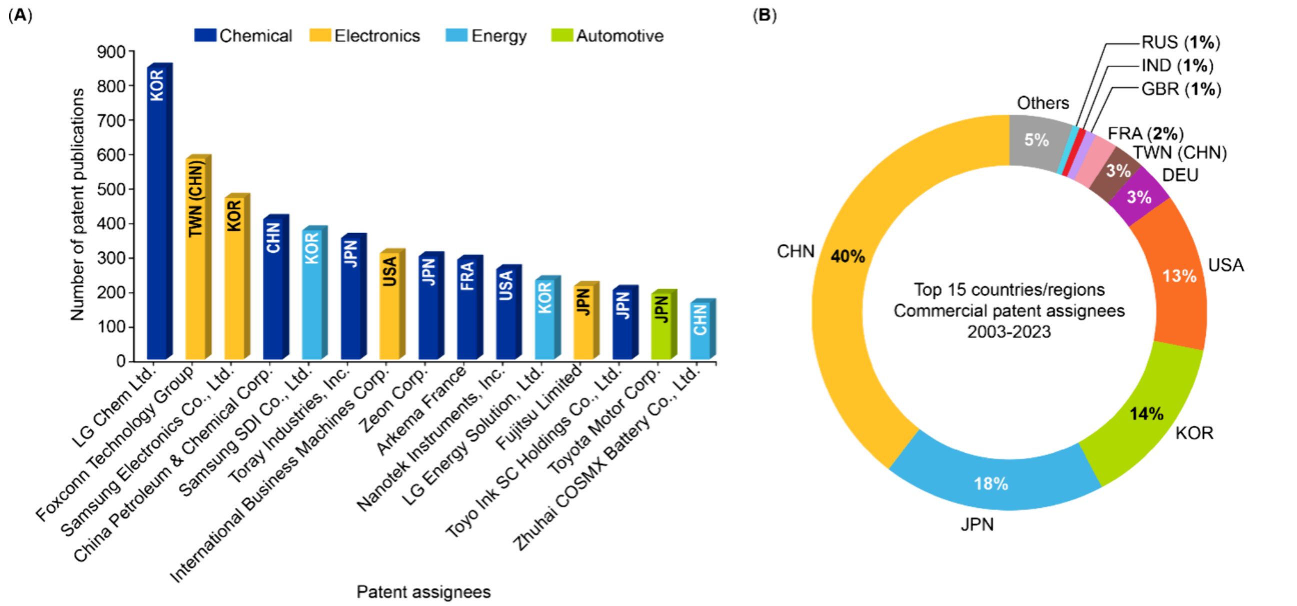

Figure 4. (A) Leading patent assignees among commercial organizations in the field of CNT-related research. Bars have been colored to indicate industry type. (B) Geographical distribution of commercial patent publications. Data includes patent publications for CNT-related research from CAS Data Collection for the period 2003 to 2023.

detail in the "Applications of carbon nanotubes" section. We have chosen to include data from 2023 however the data is partial (encompassing the months January to July) and its publication numbers should not necessarily be taken as part of a publication trend.

Analysis of the geographical distribution of publications indicates that commercial and noncommercial entities from China (CHN) appear to dominate in the field of CNT-related research with the total number of publications exceeding those from the United States (USA), South Korea (KOR) and Japan (JPN) combined (Figure 2B). Other key countries/regions include India (IND), Iran (IRN), Germany (DEU), Russia (RUS), Taiwan (TWN), France (FRA), the United Kingdom (GBR), Italy (ITA), Canada (CAN), Spain (ESP) and Brazil (BRA). The patent-to-journal ratios for these countries/regions indicates that most of them have a higher proportion of journal publications, the exceptions being South Korea and Japan, which have a slightly higher proportion of patent publications. India and Iran on the other hand, have an overwhelming number of journal publications as compared to patent publications. Journal and patent publications originating from organizations based in China (CHN) have been on the rise consistently over the years (Figure S6). Besides China, India (IND) and Italy (ITA) exhibit a steady albeit muted increase in journal publications while those from Korea (KOR) and Iran (IRA) have remained more or less consistent (Figure S6A). In terms of patent publications, Korea (KOR) shows a marked increase over the years while USA and Japan (JPN) have held steady over the years (Figure S6B).

Next, we identified leading organizations in CNT research by considering both the total volume of publications and average number of citations per publication, the latter parameter being a rough representation of the influence exerted by the institution in this field. These institutions are fairly well distributed geographically, with representation from the United States, Singapore (SGP), the United Kingdom, China, Japan, and South Korea (Figure 3) - though 60% of the leading 15 organizations originate from USA. Stanford University was identified as having the highest average number of citations per publication, with examples of highly cited publications including the use of CNTs in chemical sensors,[31] field emitters,[32] and FETs.[33] Other important research organizations identified using this method include the University of Pennsylvania (with highly cited papers on topics including CNT-containing nanocomposites[34−36] and CNT solubilization in water[37]), and Northwestern University (with highly cited papers on sorting CNTs[38] and amino-functionalized CNTs[39]).

Our analysis indicates that the top commercial assignees for CNT-related patents include companies involved in the electronics, chemical, battery, and automotive industries (Figure 4A). Patents from the leading commercial assignees discuss an array of diverse subjects, distributed across a wide range of applications. For example, recently published patent applications assigned to LG Chem, the leading patent assignee, include a reactor to synthesize CNTs that uses a nozzle to feed reactant which generates a spiral flow,[40] a mixed metal/inorganic/CNT catalyst for CO_2 electrochemical reduction,[41] and a composite Si/CNT anode for lithium secondary batteries.[42] Recently published patent applications assigned to Foxconn Technology Group (also known as Hon Hai Precision Industry Co., Ltd.), the top electronics manufacturer in terms of assigned CNT patents, include a Si/CNT lithium battery anode,[43] an electrically modulated light source based on aligned CNT films,[44] and a CNT-based transistor structure.[45]

Geographical distribution of commercial patent assignees indicates a sizable contribution by China (40% of the total), Japan, South Korea, and the US. Countries/Regions with smaller degrees of contribution include Germany, Taiwan, France, United Kingdom, India and Russia (Figure 4B).

■ CARBON NANOTUBE SYNTHESIS

Several methods are known for synthesizing CNTs. In the arc-discharge method used in the initial experiments in the early 1990s, a high DC current is passed between carbon electrodes,

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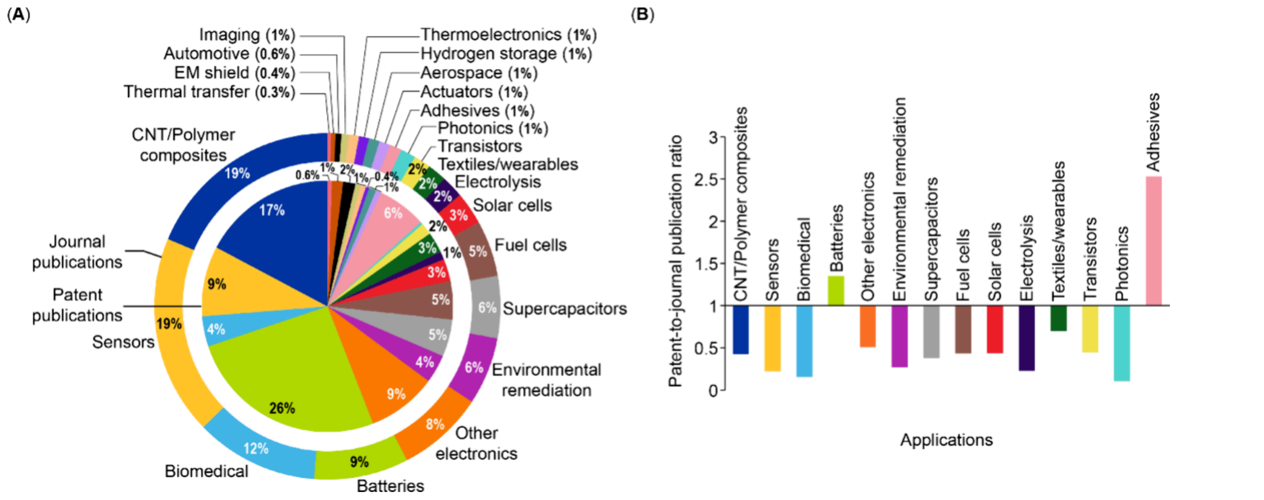

Figure 5. (A) Pie chart showing the distribution of publications related to individual CNT applications for journals (outer) and patents (inner). (B) Ratio of patent to journal publications for selected applications. Data includes journal and patent publications in the field of carbon nanotubes from the CAS Content Collection for the period 2003−2023.

leading to carbon evaporation and the formation of MWCNTs. The first demonstration of controlled SWCNT synthesis also used arc discharge, with the inclusion of a transition metal catalyst. Later studies showed that ablating a carbon target using a high-powered laser could also be used to form CNTs.[7−9]

However, as shown in Figure S7, the most common method for synthesis of CNTs over the last two decades is based on CVD,[46,47] wherein passage of a carbon-containing precursor gas over a substrate in a reactor allows for reaction between the precursor and the surface leading to gradual buildup of CNTs. The substrate can incorporate a catalyst, commonly nanoparticles made of Fe, Ni, Co, or combinations of these metals, which can nucleate CNT growth and allow it to proceed at lower temperature. A catalyst is generally not needed for MWCNT synthesis, but is needed to make SWCNTs, since the size and composition of the catalyst determines the initial stages of CNT growth, which determines the diameter and chirality of the final nanotube.

The overwhelming prevalence of CVD can be attributed to advantages associated with it, such as scalability, industrial maturity of the technique, and adaptability and flexibility through the form factor of the reactor. An example of this adaptability is floating catalyst CVD, where catalyst particles are suspended in a stream of gas containing a hydrocarbon precursor, which decomposes and forms CNTs on the catalyst surface. The floating catalyst process can be adapted to generate a range of CNT macrostructures, including films, fibers, arrays, and sponges.[48]

In plasma-enhanced CVD (PE-CVD),[49,50] a plasma above the substrate is used to partially dissociate precursor molecules before surface reactions take place. This allows CVD to be achieved at lower temperatures, which allows better control of CNT properties in some cases.[51,52]

A significant challenge in CNT synthesis by CVD is achieving the required selectivity for chirality, diameter, and other structural parameters for a given application. The CVD process has several parameters that can be used to control structure, including the catalyst type, catalyst morphology (thin film, supported, nanoparticle), gas precursor composition, template, and reaction temperature.[53]

Our analysis indicates that the frequency of publications associated with synthesis of CNTs began to decline around 2006 (Figure S7B). However, while the number of studies on the synthesis of CNTs in general appears to be decreasing, there are some areas of continued active research. For example, some emerging CNT applications require very precise control over chemical and electrical properties, so tuning synthesis parameters to achieve those properties continues to be an active area of research.[54,55] One particular area of focus is controlling the distribution of chirality. For example, in electrical applications, high selectivity for a particular chirality is needed because the chirality controls the bandgap of a SWCNT. The most widely studied parameters to control chirality involve the catalyst,[56] in particular varying the catalyst particle size, crystal structure, support, and melting point.[57,58]

Large-scale pyrolysis of methane and natural gas has seen growing interest recently as a possible solution for lowering CO_2 emissions associated with industrial and residential energy supply and generation of hydrogen for industrial feedstocks.[59,60] In this process methane, natural gas, and other hydrocarbons are converted into hydrogen and solid carbon in a high temperature, nonoxidizing environment.

The economic viability of this process, sometimes known as the "turquoise hydrogen" process, would be greatly improved if a high-value end use for the generated solid carbon could be identified. For this reason, there has been significant research efforts by academic and industry research groups to develop catalysts and/or catalyst processing methods that can sustainably generate CNTs as the carbon coproduct from high volume methane pyrolysis. The ability to generate CNTs through the catalytic pyrolysis of methane is well known.[61] However, the main practical challenge to scaling up these processes, which is a general obstacle for any catalytic pyrolysis process regardless of the carbon morphology generated, is the deactivation of catalysts over time due to the buildup of solid carbon on the catalyst surface. An effective method for catalyst regeneration is to oxidize the carbon layers. However, in this case, oxidation cannot be used since it involves both the destruction of the CNTs and the generation of additional CO_2.

However, processes have been demonstrated to aid in the regeneration of CNT-producing methane pyrolysis catalysts and recovery of the formed CNTs. Some of these can be

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implemented *in situ*, without removing the catalyst from the pyrolysis reactor, enabling varying degrees of continuous or semicontinuous operation. One example is the use of a fluidized bed reactor with an alumina-supported Fe catalyst, where CNTs were dislodged periodically using a steam/argon mixture.[62] Another route involves the development of catalysts that generate CNTs which can be more easily dislodged from the catalyst surface.[63]

APPLICATIONS OF CARBON NANOTUBES

Owing to the unique combination of material properties, CNTs are used in a wide variety of applications where they play critical functional roles.[64] Figure 5 shows the distribution of CNT applications under development referenced in journal and patent publications.

The largest fraction of CNT publications focused on their use in composites,[65] including polymer-matrix composites (Figure 5A). The structure of CNTs gives them strength along their longest dimension, while their length allows them to act on macroscopic length scales. In addition, the ability to functionalize the exterior of CNTs either covalently or noncovalently can be used to make them compatible with a variety of materials. The electrical and thermal conductivity of CNTs allows them to act as functional components and not just as structural components. Composites are used in many products on both large and small scales; their ubiquity and the useful characteristics of CNTs in them can account for their large fraction in CNT applications. Some of the applications for CNTs in composites include their use in batteries,[66,67] in biocompatible hydrogels for heart cell growth,[68] for use with a glycidyl polyketone to form nonconductive layers,[69] to increase strength and wear resistance in composites with high-density polyethylene,[70] to absorb high-frequency electromagnetic waves (CN117042427A[71]), and as components in heat-resistant aerogels (CN114275770[72]). One caveat with the analysis of composites is that the term "composite" can refer to both a direct use of CNTs for their physical properties (i.e., structural composites where CNTs are incorporated into a matrix to provide mechanical strength, or electrical or thermal conductivity) and a more general structure type in which CNTs are combined with other components (such as their use as catalyst supports); classification as a composite thus may not exclude the use of CNTs in other applications.

The largest fraction of patent publications on CNT applications was for use in batteries (Figure 5A). The length of CNTs and their conductivity makes them useful as molecular wires, and their strength could allow them to reduce the expansion and contraction of battery materials during charging/discharging, which leads to component separation and battery failure, or to form a barrier to dendrites, preventing short circuits and their associated hazards. These characteristics motivate the use of CNTs in batteries such as (with ZnO and carbon felt) in lithium-ion (Li-ion) battery electrodes resistant to lithium dendrite formation,[73] and materials such as graphene networks, $NiCoO_2$, MnO_2, nitrogen-doped carbon, and cobalt−iron metal−organic frameworks in biocompatible and hybrid zinc or in zinc-air batteries.[74−76] One feature of note is that battery applications represent a much higher fraction of patent publications compared to journal publications (more journal publications are devoted to CNT-containing sensors than CNT-containing batteries). Many of the patents for batteries originate from China or South Korea; a push to transition from internal combustion to electrical power for transportation, and the large consumer electronic industries in those nations likely motivate battery development and commercialization. For recyclable batteries, laboratory-scale battery manufacturing and testing can differ significantly from the methods required to produce batteries on larger scales, making the type of research performed for journal publication difficult to directly implement on large production scale, perhaps decreasing commercial interest in CNT exploratory research for batteries.[77] The ratio of patent to journal publications in the use of CNTs in batteries (Figure 5B) is consistent with other battery research; for example, in Li-ion battery recycling, patent publications exceeded journal publications by nearly a factor of 3.[78] CNTs are also useful components for supercapacitors.[79] Supercapacitors generally cannot store as much energy as batteries but provide higher specific power[80] and thus enable power storage for applications not amenable to batteries. CNTs function in similar ways in both systems−their high specific surface areas, conductivities, and electrochemical stabilities allow them to rapidly gather and transport charge. The significant fraction of publications discussing the use of CNTs in supercapacitors indicates interest both in supercapacitors and in CNTs.

Sensors incorporating CNTs are a significant area of research, comparable to CNT/Polymer composites (Figure 5A). The strength and aspect ratio of CNTs make them able to transmit physical forces, while their electrical conductivity makes them useful at converting and transmitting stimuli. In addition, CNTs have a large specific surface area, increasing their susceptibility to external changes. A variety of sensors using CNTs have been assembled, such as a pressure sensor using conductive polymers, CNTs, and poly(dimethylsiloxane) (PDMS) (CN2023116972905A[81]), CNTs supported on polyurethane for use as a triboelectric generator, movement sensor, or chemosensor,[82] with superhydrophobic surface as a flexible airflow sensor,[83] in concert with nickel triphenylene complexes as a nonbiological glucose sensor,[84] and as nanoresistors to detect disinfectants.[85] MWCNTs have been used in combination with samarium to detect solvent vapors,[86] with polyaniline (PANI) to form ammonia-responsive materials,[87] with epoxy resin to form a strain-sensing conductive material,[88] and with polyurethane and polypyrrole to form a bendable pressure sensor[89] (in the latter sensor, the use of components with differing compressibilities was critical to its effectiveness). The fraction of patent publications using CNTs for sensors was smaller than the corresponding fraction of journal publications, making up 19% of journal publications but only 9% of patent publications (Figure 5). One possible explanation is that sensor fabrication on laboratory scale may require more labor to assemble or more expensive materials to be readily transferable to large scale; alternatively, laboratory-conceived sensors may either be proofs of concept for sensor design or may utilize target stimuli less amenable for further development.

Other applications of CNTs make up significant fractions of the publication record. Biomedical uses of CNTs make up 12% of journal publications, though only 4% of patent publications. CNTs have been used as a reinforcing material for drug-delivering stents made with biocompatible polymers (WO2014186532A1[90]), as a conductive material with poly(3,4-ethylenedioxythiophene) (PEDOT) used as a coating for nerve catheters (CN2023116983475A[91]), as an antibacterial and structural material for bone implants.[92,93] CNTs have also been used as delivery vehicles for drug and gene moieties.[94,95]

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While CNTs are readily functionalized, allowing their surface chemistry and charge to be altered appropriately for bioavailability, they can also exhibit toxicity depending on their purities, sizes, shapes, and whether they are integrated into macroscale forms,[96] and may require dispersion or solubility in water;[97] the properties necessary for the desired activities may lead to side effects, making them less commercializable. The safety of CNTs generally is an active area of research and policy debate.[98]

Besides sensors, CNTs have also been incorporated into other electronic materials accounting for 8% and 9% of journal and patent publications, respectively. As noted earlier, the ability to functionalize CNTs (with the purpose of modifying their electronic and chemical properties) is important. The orientation and morphologies of CNTs can also be manipulated to achieve the desired device properties, but the manipulation of these properties is often manual and may require significant optimization to be produced on larger scale. The relative amounts of patent and journal publications indicate that the benefits of CNT design likely outweigh impediments to their commercial uses. Patents are an indication of the amount of commercialization activity in a certain field, since the primary reason for filing patents is to protect commercial interests. So, in other words, despite challenges to their use, patent frequency suggests that the research community believes they have a high degree of commercial potential. Meanwhile, journal publication rates indicate the level of research being conducted at the academic stage. Many other fields tend to show this lag between academic-oriented and commercial research.[6] Some of the potential uses of CNTs in this area include as components in FETs (WO2022040565[99]) for instrument amplifiers, with bamboo-derived fibers, phenolic resins, and cobalt in a layered electrode material,[100] and in a conductive fabric.[101]

Environmental remediation is a significant application for CNTs, with roughly 6% of journal publications and 4% of patent publications discussing it (Figure 5A). The high surface area and conductivity of CNTs are important properties, as they make CNTs available for absorption and for mediating interactions with pollutants. The ability to modify CNTs allows them to be tailored for other cocatalysts or for reaction with pollutants. For example, lanthanum- and iron-modified CNTs have been disclosed for environmental use (CN2021113842883A[102]), while a lanthanum–gadolinium-iron oxide-perovskite reinforced with CNTs showed improved photocatalysis of the degradation of phenol red over the CNT-free perovskite.[103] A CNT membrane was used to activate MnO_2 for oxidative degradation of water pollutants.[104] Journal articles describing the use of CNTs in environmental remediation are significantly more numerous than patent publications (Figure 5B); the disparity may be due to the lack of economic incentive in environmental remediation which makes incremental improvements or improvements with more expensive materials difficult to implement. Even though SWCNTs can conduct electrons well and have high surface area, enabling improved reactivity and utility, their expense may make their use in environmental remediation difficult because the increased costs cannot be recovered by increased prices.

Besides batteries, the use of CNTs in adhesives is the only other application with a high patent to journal ratio (Figure 5B), which suggests considerable commercial interest in this area. The shape of CNTs could allow them to become entangled; alternatively, functionalized nanotubes could react with surfaces with their strength making them strongly adherent. For example, CNTs were used to reinforce PDMS dry adhesive pads;[105] the CNTs improve the stability of the pads and diffuse concentration of charge at the PDMS surface which leads to surface deformation and loss of conformity. Alternatively, a poly(ethylenimine), CNT and epoxy resin material was used as an adhesive.[106] Here, the nanotubes improve the strength/mass ratio of the material, while adhesion depends on the fraction of poly(ethylenimine) used. Selected patents describe the use of CNTs for strengthening concrete (RU2669835[107]) and the use of a *trans*-stilbene/maleimide polymer-modified CNT to make an adhesive with improved thermal conductivity (CN2023116970357A[108]).

The co-occurrence of SWCNTs versus MWCNTs for different applications is shown in the form of a Sankey graph (Figure 6A). The choices of which type of CNT to use in

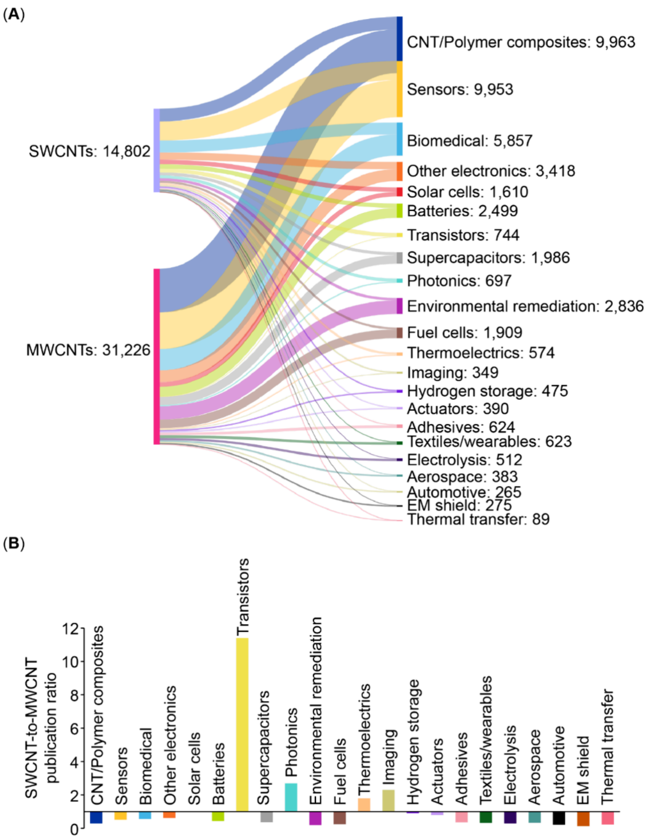

Figure 6. (A) Co-occurrence of single-walled carbon nanotubes (SWCNTs) and multiwalled carbon nanotubes (MWCNTs) with selected applications. (B) Ratio of SWCNT to MWCNT publications for selected applications. Data includes journal and patent publications in the field of CNTs from the CAS Content Collection for the period 2003–2023.

different circumstances depends on the necessary properties and the costs of the CNT chosen. Both SWCNTs and MWCNTs can be either semiconducting or metallic, with variable thermal and electrical conductivities.[109,110] SWCNTs may offer advantages in particular circumstances. SWCNTs are less structurally heterogeneous, allowing more reliable functionalization. In addition, SWCNTs have predictable Raman and near-IR absorptions, with their near-IR absorption

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in a region at which tissue is most permeable, making them better suited for use in biological and medical sensing applications, The diameters of CNTs may differ significantly; SWCNTs are roughly 1 nm in diameter, while MWCNT can have diameters between 2 and 100 nm.[110] Purified SWCNTs with reduced metal content can be obtained for 2000 USD/kg though a 99% SWCNT content grade with <0.1% metal content is available, and MWCNTs can be obtained for 100 USD/kg.[111−114]

As shown in Figure 6B, for most of the applications of CNTs, MWCNTs are predominantly used, likely because they are significantly less expensive than SWCNTs. Publications discussing solar cells, hydrogen storage, and actuators, however, use SWCNT and MWCNT at similar frequencies. SWCNTs are also used fairly extensively in biomedical applications, where their effective absorption and fluorescence properties at near-IR wavelengths make them amenable to tissue penetration and potential use in living things.[115] SWCNTs are likely to have more predictable functionalization behavior and toxicities, important for biocompatibility.

Sensors and electronics use SWCNTs less frequently than MWCNTs but at significant frequencies. SWCNTs are likely to have more predictable electronic behavior, while the presence of a single structure instead of multiple structures allows them to be physically manipulated and aligned, necessary for use in field-effect transistors and other small device components.[109] On the nanometer scale, SWCNTs are likely the only sufficiently predictable CNT that can be used, and on small scale or for proof of principle, well-defined CNTs are important for understanding the behavior of the devices made using them. The single walls of SWCNTs makes the paths of electrons through the CNT more responsive to external stimuli, and thus may be more effective as sensors. However, MWCNTs can be used in gas sensors,[109] and their reduced cost may make them useful alternatives to SWCNT in nonbiological sensing. Transistors and photonics use SWCNTs almost exclusively, likely because of the strong relationship between nanotube structure and electronic behavior and the need for consistent electronic behavior. Publications discussing composites show the use of SWCNTs and MWCNTs at similar rates. Many of the functions that are needed for composites can be performed both with SWCNTs and MWCNTs; both have similar Young's moduli and both can be thermally and electrically conductive.[110] The choice of SWCNT or MWCNT in composites may depend mainly on the fraction of nanotubes needed to impart the desired properties to the composite without compromising its other properties.[116]

In Figure 7, the landscape of emerging trends in the field of CNTs was mapped by identifying concepts that were found the most frequently in literature from 2020 to 2022, or that increased significantly in usage in the literature during that time using a NLP-based method.[4] These emerging concepts were grouped into the three major areas of properties, materials, and applications, with further subcategorization within each area. Within properties, two notable concepts that were frequently found in CNT literature were *thickness* and *thermal conductivity*. Thickness appears to primarily occur in publications wherein CNTs are incorporated in microwave absorbing and electromagnetic interference (EMI) shielding composites, to describe the optimal or tested physical thickness of the shielding material.[117−119]

Thermal conductivity is commonly used in the context of using CNTs to improve the thermal properties of composite materials, and for that reason there is no dominant application that results in the high use of this term. Recently, these composite materials have included phase change materials (PCMs), which are used to store energy in the form of latent heat.[120,121] In these materials, CNTs are incorporated with a bulk phase change material, such as paraffin, where the high thermal conductivity of the CNTs reduces the amount of time needed for the phase change material to take up and release thermal energy. A major advantage to using CNTs for this purpose is that they can improve thermal conductivity in phase change materials significantly at relatively low concentrations, as shown in a recent study where adding 5 weight% CNTs increased the thermal conductivity of a polyurethane PCM by 2.3 times.[122]

In addition, CNTs are used in solar water evaporation devices which convert seawater to freshwater,[123] where low thermal conductivity is desirable to prevent heat loss through conduction. In these devices, CNTs are used primarily to take advantage of their high photothermal conversion efficiency. Recently, it has also been demonstrated that aligned CNTs can be incorporated into self-healing polymer matrices, providing high thermal conductivity to intrinsically self-healing materials;[124] both are critical properties in thermal interface materials used in high-power and dynamic devices.

The properties associated with the fastest growth in CNT-related publications are *brittleness* and *exergy*. Prominent examples of the use of the term *brittleness* include reinforcing materials with CNTs to reduce their intrinsic brittleness, including hydrogels[125,126] (primarily for use in sensors), poly(lactic acid) (PLA)[127] (enabling the use of this biodegradable polymer for mechanically demanding applications), epoxy resins,[128,129] conjugated microporous polymer membranes,[130] and silica aerogels.[131] Other examples of brittleness relate to the use of CNTs as replacements for brittle, traditional materials used in battery components and other applications.[132] Brittleness also appears in the context of strain sensors which combine brittle materials and CNTs in composites,[133,134] where cracking of the brittle material in response to strain improves the piezoresistive response, resulting in a higher gauge factor.

The majority of references where the concept of exergy is referenced involve the use of CNTs as thermal conductivity additives in nanofluids used for heat transfer,[135,136] specifically in photovoltaic/thermal (PVT),[137,138] solar thermal,[139] and combined PVT/PCM[140] applications, where exergy efficiency is used as a measure of the performance of these fluids.

In the "Materials" branch, terms with an especially high growth rate (>1.5 fold increase in publications over 2020−2022) include poly(butylene adipate-*co*-terephthalate) (PBAT), ciprofloxacin, biochar, cellulose nanofibers, and MXene nanosheets. This branch includes both materials that are often used in combination with CNTs as well as materials that are detected or removed using CNTs.

PBAT is a biodegradable polymer which has been recently used to make electromagnetic shielding materials in combination with CNTs.[141−144] Because of its mechanical strength, PBAT has also been combined with PLA by melt-blending to make PBAT/PLA/CNT composites.[145,146]

Ciprofloxacin is a commonly used antibiotic which is difficult to remove via conventional wastewater treatment methods, and has been detected at significant levels in the

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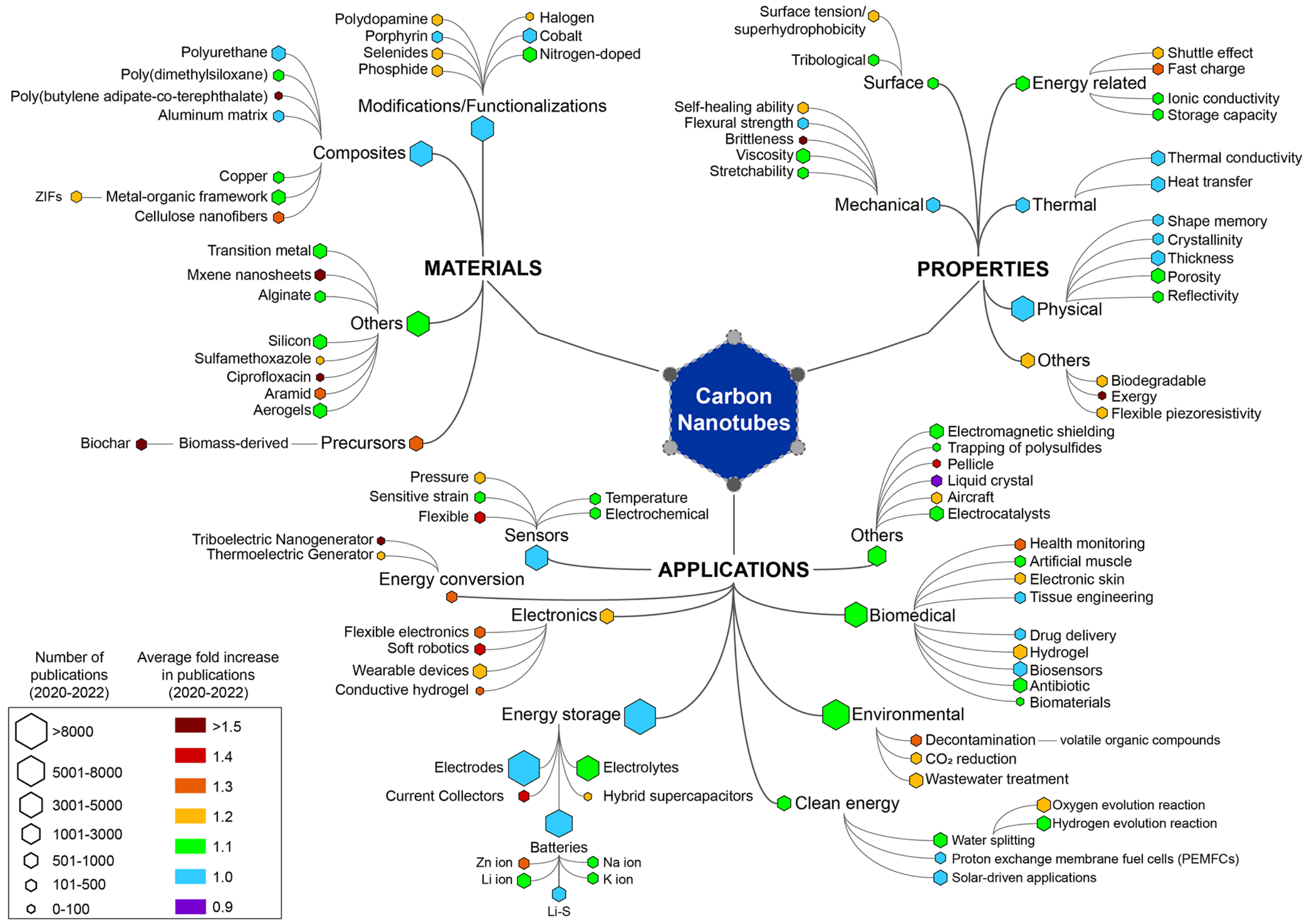

Figure 7. "Trend landscape" map of emerging concepts in CNT publications resulting from NLP analysis of more than 265,000 publications. Total number of publications from 2020 to 2022 and relative publication growth rate shown by size and color of each node, respectively.

environment.[147] It is toxic to certain aquatic organisms,[148] and can interact with bacteria in the environment, contributing to a rise in antibiotic resistant pathogens and impacting wastewater treatment processes.[149] CNTs can be used to remove ciprofloxacin from water through adsorption and catalytic processes. When used as adsorbents for wastewater, CNTs can be modified with magnetic nanoparticles such as ferrites[150] or Fe_2O_3,[151] allowing them to be separated downstream using a magnetic field. Examples of catalytic removal of ciprofloxacin include those where CNTs perform a catalytic function (through the generation of OH radicals[152]), and where CNTs are combined with other catalytic materials,[153,154] or are used to support oxidizing compounds in ultrasound-assisted oxidation processes.[155,156]

The co-occurrence of biochar with CNTs seems to be driven primarily by studies which compare the performance of the two materials for their ability to adsorb or otherwise treat environmental pollutants,[157,158] reflecting the growing importance of this application. To a lesser extent, biochar and CNTs have been combined in composites for heavy metal adsorption and energy storage;[159,160] biochar can also be used as a precursor for CNT production.[161,162]

Cellulose nanofibers are similar in size and shape to CNTs, and have recently been combined with CNTs in a variety of applications. These include conductive, hydrophilic aerogels made using a mix of cellulose nanofibers and CNT,[163,164] hydrogels made using a mixture of 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO)-oxidized cellulose nanofibers and CNTs.[126] Cellulose nanofibers and CNTs can also be combined in composite electrodes, where 3D networks of the two materials are used as a support for active materials.[165,166]

The rapid growth of publications involving both MXenes and CNTs is not unexpected, since MXenes have been a rapidly growing field of research since their discovery in 2011.[167] They have been tested in a wide range of applications, including energy, catalysis, and biomedicine. Sensors has been a relatively recent application area for MXenes, with 2017 having been identified as their first significant use in this field.[168] A recent example is a piezoresistive strain sensor based on a composite thermoplastic polyurethane (TPU)/CNT foam with a layer of MXene ($Ti_3C_2T_x$) deposited on its surface.[169] The deformation and cracking in both the foam and MXene layers results in a change in resistance per unit strain and is much higher than the sum of analogous sensors made using only a TPU/CNT foam, or a TPU foam/MXene sensor without CNTs. In this example, the CNTs and MXenes are not combined on a microscopic level, but their complementary properties lead to a synergistic effect.

CNTs can also be combined with MXene nanosheets on a nanoscale level, making 2-D/1-D composites composed of alternating layers of individual MXene sheets/CNTs, for

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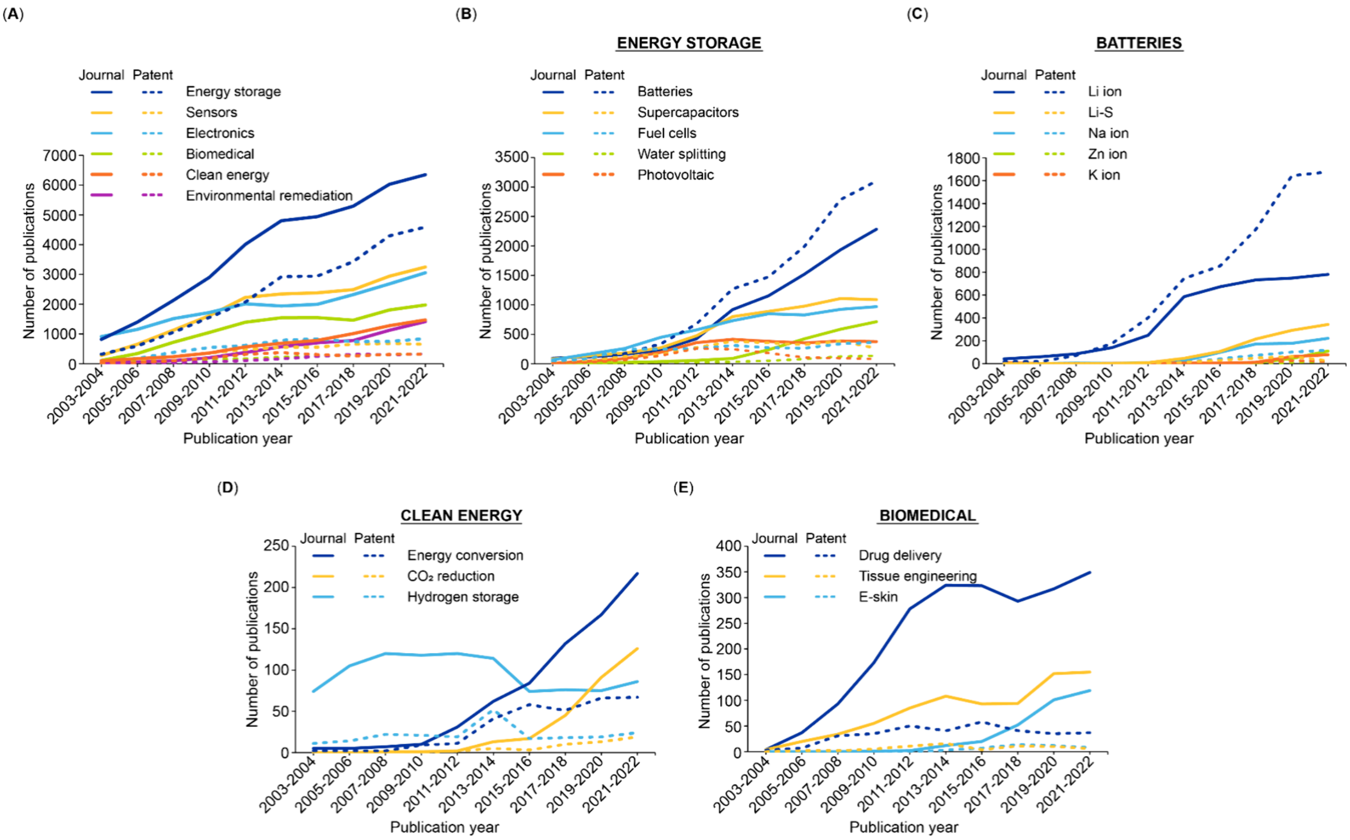

Figure 8. Time trends for selected concepts and concept groups from Figure 7. Growth in journal and patent publications for (A) broader emerging applications, and applications specific to (B) energy storage, (C) batteries, (D) clean energy and (E) biomedical. Data includes patent and journal publications in the field of CNT-related research from the CAS Content Collection for the period 2003−2022.

applications including battery electrodes[170,171] and separators,[172] supercapacitors,[173] EMI shielding,[174] water splitting,[175] and sensors.[176] In these materials, CNTs are used for a variety of reasons, including to control the spacing between MXene sheets and to prevent sheets from restacking[170,177] (to improve ion transport in battery electrodes,[178] for example), to improve mechanical properties such as tensile strength,[171,179] electrical conductivity,[175] and electromagnetic shielding and absorption.[180] CNTs can also be used as a strategy for embedding metallic nanoparticles between MXene sheets.[181]

Turning to the "Applications" branch, energy storage is the most active area in terms of total number of publications, with battery research driving much of that growth, as shown in Figure 8B. Other notable application areas with a large number of publications in 2020−2022 include sensors, environmental, and biomedical (Figure S8). Fewer total publications, but more rapid growth, is seen in electronics and energy conversion applications. Full time trends of journal and patent documents in these areas are shown in Figure 9A.

The breakdown of CNT-related publications for different battery chemistries is shown in Figure 8C. Li-ion batteries dominate, but Zn-ion batteries represent the highest growth. In Zn-ion batteries, prominent examples of the use of CNTs are in the cathodes of aqueous Zn-ion batteries, in combination with $KV_3O_8{\cdot}0.75H_2O$ (KVO),[182] $ZnMn_2O_4$ (ZMO),[183] M phase VO_2,[184] $Na_xV_2O_5{\cdot}nH_2O$(NVO),[185] and V_2O_5.[186] In these cases, the role of the CNT is to form a high surface area, mechanically resilient, flexible, and electron-conductive scaffold that supports these materials. The goal of improving these properties is to improve the rate capability and capacity retention after repeated cycling. CNTs are also used to enable Zn metal anodes, either as a way to control Zn deposition, preventing the formation of dendrites,[187] or as a scaffold for Zn nanosheets.[188,189]

Research in triboelectric nanogenerators and thermoelectric generators are two other emerging applications of CNTs (represented as "energy conversion in Figure 8D). Triboelectric nanogenerators are devices which can generate electrical energy from many different types of motion, including human motion, or the motion of plant leaves in the wind.[190] The origin of this electrical energy is charge separation that takes place during the mechanical interaction of two surfaces. In this application, CNTs are used to increase the surface charge density,[191−193] thereby increasing the output power of the device. CNT-containing triboelectric nanogenerators have many possible applications, including self-powered sensors, or providing power to wearable devices.[194,195]

Hydrogen storage shows a significantly different trend compared to the other energy topics, with generally flat or negative growth since 2010. There appear to be multiple reasons for this. One reason is the difficulty in obtaining reproducible and accurate quantitative measurements of hydrogen storage capacity,[196,197] the other being that it is now thought that pure CNTs have a fairly low gravimetric storage capacity for hydrogen at room temperature.[197] This is below the initial US Department of Energy goal set in 2010 of 6.5 wt % for automotive H_2 storage applications and which was later reduced to 5.5 wt % in 2015. However, work continues on

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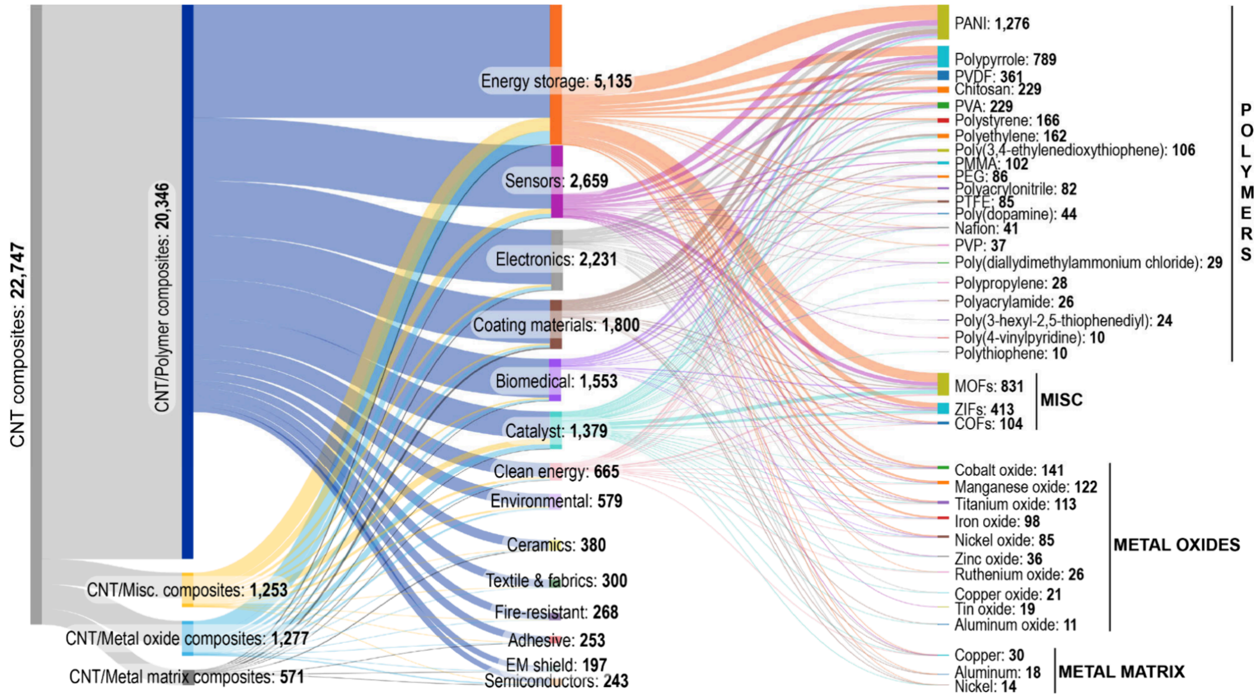

Figure 9. Breakdown of CNT composites based on journal publications in the field of CNTs from the CAS Content Collection for the period 2003−2023. The second and third columns show types of CNT composites and applications in which they are frequently utilized, respectively. The column on the extreme right shows individual polymer, metal oxide and metal matrix materials as well as materials such as metal−organic frameworks (MOFs), zeolite imidazole frameworks (ZIFs) and covalent organic frameworks (COFs) grouped under CNT/Misc. composites.

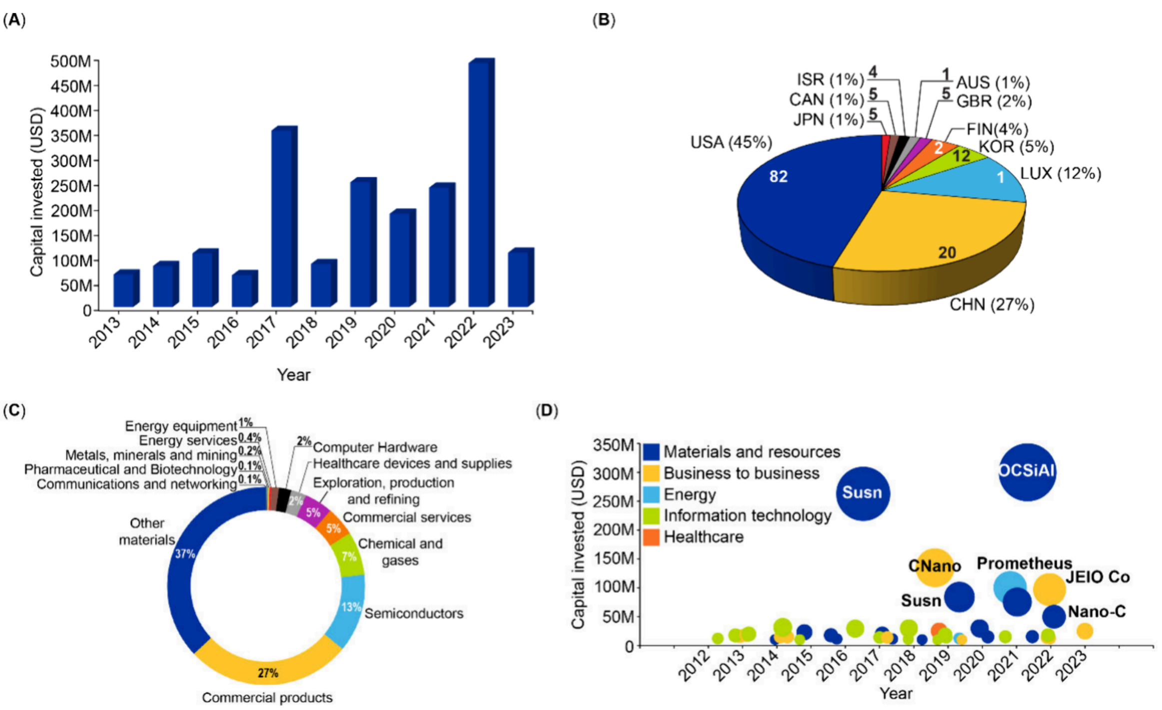

Figure 10. Commercial interest in CNTs. (A) Capital invested in the field of CNTs over 2013−2023. (B) Geographical distribution of capital invested in the area of CNTs over 2012−2023. Whole numbers (without units) indicate number of companies based in that geographical area. (C) Breakdown of capital invested across primary industry groups in the area of CNTs over 2012−2023. (D) Top 50 deals in the area of CNTs over 2012−2023 categorized by primary industry sector.

the use of modified CNTs for hydrogen storage, for example CNTs loaded with metals or heteroatoms such as N.[198,199]

A final application to highlight in Figure 7 is the use of CNTs in flexible sensors and electronics. CNTs are used here primarily for their electrical properties, which can impart piezoresistive, piezocapacititve,[200] and other properties useful for sensor applications. This can involve incorporating CNTs into matrices, including hydrogels,[126,201] or combining them with polymers (for example, in a polyurethane/CNT yarn with hierarchical structure[202]). Flexible sensors can also be fabricated by printing CNT-containing ink onto flexible substrates.[203] The major emerging application in these cases is monitoring human motion, either through wearable sensors or electronic skin,[204] as shown in Figure 8E. The other prominent biomedical application of CNTs is drug delivery.[205] However, publication trends in this area (Figure 8E) indicate this is a relatively mature field of research.

As shown in Figure 8D, another emerging application for CNTs is in the electrochemical reduction of CO_2. This can be used as an approach for carbon capture that simultaneously generates a variety of useful chemical products. CNTs can be used in several ways related to catalyzing this process, including as a scaffold for supporting single-molecule metal based catalysts,[206−208] covalent organic frameworks (COFs),[209] and pipet-like bismuth nanorods.[210] CNTs, particularly when they are doped with nitrogen or other atoms, can also catalyze CO_2 reduction, acting as metal-free electrocatalysts.[211−213]

To better understand the use of CNTs in composites, we have broken down the types of composites by the matrix material (left), application (center), and the most commonly used matrix substances (extreme right) and showed their co-occurrences in a Sankey graph (Figure 9). This data reflects the common use of polymers in CNT composites, for a variety of applications. Among these, the use of conductive polymers including PANI, polypyrrole, and others in energy storage and sensor applications is especially prominent. Examples include the use of a PANI/SWCNT composite in Al-ion batteries,[214] a combination of polypyrrole and CNTs to enable the use of black phosphorus in Li-ion battery anodes,[215] and the use of CNTs and polypyrrole in aqueous Zn-ion battery cathodes.[216] A similar breakdown of matrix material (left), application (center), and commonly used matrix substances (extreme right) with respect to patent publications can be found in the Supporting Information (Figure S9).

When CNTs are used as additives to improve the mechanical, thermal, or electrical properties of a polymer, a key to effective incorporation of the CNTs is preventing aggregation of the CNTs and maximizing their interaction with the polymer matrix, which can be done through functionalization of the CNTs.[217,218]

This analysis also highlights the frequent use of MOFs in energy storage applications. A recent example of this is the use of a cerium-based MOF/CNT composite separator in a Li−S battery.[219] Here, the CNT structure provides electrical conductivity and is an effective physical barrier for polysulfide shuttling, while the MOF catalyzes the breakdown of larger polysulfides into Li_2S. In another example, MWCNTs were used as a template for the growth of NiCo-based MOFs, improving their performance as supercapacitor electrodes.[220]

■ INVESTMENT INTEREST IN CARBON NANOTUBES

Investment data from Pitchbook[221] is indicative of growing commercial interest in CNTs over the past decade (Figure 10A). In terms of geographical distribution, almost half of the capital invested (45%) between 2013 and 2023 appears to be in 82 companies located in the USA (Figure 10B), followed by China. Luxembourg (LUX) accounts for 12% of capital invested, which can be attributed to OCSiAl, largest known manufacturer of commercially available CNTs (Tuball). Other countries/regions showing commercial interest in CNTs include South Korea, Finland (FIN), the United Kingdom, Australia (AUS), Israel (ISR), Canada and Japan.

A breakdown of primary industry groups indicates interest in CNTs across a wide range of industry types including semiconductors, energy and biomedical (Figure 10C) with the latter accounting for a smaller fraction. Among the largest 50 deals in the area of CNTs over the past decade, the majority appear to be in the material resources, information technology and business to business segments (Figure 10D). Energy and healthcare sectors account for very few deals of large magnitudes. In the first quarter of 2022, OCSiAl received the largest capital investment of nearly 300 million USD from a group of venture capitalists.[221] Similarly, significant capital investments, but of a smaller magnitude, were also received by JEIO Co and Nano-C in 2022. JEIO Co, a South Korean company, manufactures CNTs and specializes in their applications in Li-ion batteries while Nano-C appears to specialize in SWCNT manufacturing with possible use/applications in sensors and electronics. In addition, Nano-C also appears to manufacture SWCNT ink that can be deposited on the surface using a variety of printing methods.[222]

■ CONCLUSIONS

We have used natural language processing to identify the most prominent and emerging applications, associated materials, and properties of carbon nanotubes in journal and patent publications from 2020 to 2022. We have combined this with our in-depth landscape analysis to show that polymer-matrix composites, sensors, biomedical applications, and batteries have been the most prominent applications over the last 20 years. Though with high potential, as with many other materials, the use of CNTs has been linked to health and safety concerns.[223] More studies will be beneficial to further estimate the health and environmental risks, as well as to develop preventive mechanisms, along the way of advancing CNT-based nanotechnology. Among the diverse applications, the use of CNTs in batteries has a significantly higher patent to journal publication ratio, indicating relatively higher commercial R&D activity in this area.

Tracking the frequency of publications related to CNT synthesis, CVD is by far the most common method used to make CNTs, outnumbering other methods by a factor of 4−5. In general, publications related to the synthesis of CNTs have decreased since 2006. However, there is still significant research interest in achieving greater control of CNT structure through synthesis parameters.

Comparing the use of SWCNTs versus MWCNTs, we estimate that the latter are used more frequently, by a ratio of roughly 2:1, possibly resulting from a balance between cost (which favors the use of MWCNTs) and control over physical/mechanical/electrical properties (which favors the use of SWCNTs). This balance exists for each specific application, so the overall 2:1 ratio also depends on the prevalence of applications that require highly controlled properties. Certain applications such as transistors, photonics, thermoelectrics, and imaging use SWCNTs more heavily.

While there appeared to have been a plateau in the number of yearly publications on CNTs around 2014−2017, this number has generally increased since that time. To determine the trends that are driving this increase, we calculated the rate of change of specific applications, materials, and properties in CNT publications identified using NLP analysis. The results of these calculations were used to make a conceptual map of areas of high research activity involving CNTs. The main applications that appear to be driving growth include energy storage and conversion, sensors (particularly human motion and wearable sensors), environmental remediation and CO_2 reduction, and applications where CNTs are combined with other nanoscale materials such as MXenes.

Through these applications, the unique properties of CNTs, including strength, electrical and thermal conductivity, and ability for functionalization are being leveraged to address the most critical problems and promising research areas currently known. Understanding the landscape of key emerging trends in this area, specifically the fast-growing applications, properties, and materials used in combination with CNTs, will be important for researchers who are driving further growth in the use of CNTs and nanoscale materials in general.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsanm.4c02721.

Methods. **Figure S1.** An example of a citation graph (CG) showing connections between nodes. **Figure S2.** Distributions of publications and citations of the phrase "metal-organic framework" between the years 2000 and 2022. **Figure S3.** The polynomial function that approximates the actual data of publications, citations of the phrase "metal-organic framework" between years 2000 and 2022. **Figure S4.** The measures of emergence for publications and citations of the phrase "metal-organic framework" at the point of interest (year 2014). **Figure S5.** Predictions of publications and citations for the years 2020−2022. **Figure S6.** Overall (A) journal and (B) patent publication trend for leading countries or regions for CNT-related research. **Figure S7.** Methods of synthesis of CNTs. **Figure S8.** Comparison between number of publications and relative growth rate for identified emerging applications in the field of CNT. **Figure S9.** Sankey graph depicting breakdown of CNT composites−types of CNT composites, their applications and individual materials (polymer, metal oxides and others) based on patent publications in the field of CNTs. (PDF)

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ABBREVIATIONS USED

AFM, atomic force microscopy; AUS, Australia; BRA, Brazil; CAN, Canada; CHN, China; CNT, carbon nanotube; COFs, covalent organic frameworks; CVD, chemical vapor deposition; DEU, Germany; DWCNTs, double-wall CNTs; EMI, electromagnetic interference; ESP, Spain; FETs, field-effect transistors; FRA, France; GBR, United Kingdom; HiPco, high pressure carbon monoxide; IBM, International Business Machines Corporation; IND, India; IRN, Iran; ISR, Israel; ITA, Italy; JPN, Japan; KOR, South Korea; Li-ion, lithium-ion; LUX, Luxembourg; MOFs, metal−organic frameworks; MWCNTs, multiwall CNTs; NEC, Nippon Electrical Corporation; NLP, natural language processing; PANI, polyaniline; PBAT, poly(butylene adipate-*co*-terephthalate); PCMs, phase change materials; PDMS, poly(dimethylsiloxane); PE-CVD, plasma-enhanced CVD; PEDOT, poly(3,4-ethylenedioxythiophene); PLA, poly(lactic acid); PVT, photovoltaic/thermal; RUS, Russia; SGP, Singapore; SWCNT, single-wall CNT; TEMPO, 2,2,6,6-tetramethylpiperidine-1-oxyl; TPU, thermoplastic polyurethane; TWN, Taiwan; USA, United States of America; ZIFs, zeolite imidazole frameworks

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