

Patent Analysis and Trends Related to 2D Nanomaterials for Active Food Packaging [†]

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Abstract: Active food packaging technology encompasses systems that incorporate active substances into the polymeric matrix. The embedded components exhibit antimicrobial, antifungal, and antioxidant properties and are able to absorb or reduce oxygen, carbon dioxide or ethylene, thereby enhancing the quality and safety of food products. The utilization of 2D nanomaterials, such as graphene, has facilitated the advent of novel avenues for the advancement of active packaging (AP). The integration of these materials with polymers has the potential to enhance the barrier, thermal, and mechanical properties of packaging materials. The objective of this paper is to provide a comprehensive overview of patented two-dimensional materials in the field of active packaging.

Keywords: patent landscape; nanocomposites; bio-packaging; biopolymers; smart packaging; graphene; graphene oxide (GO); reduced graphene oxide (rGO)

1. Introduction

As indicated by the available data [1,2], the incidence of foodborne diseases has exhibited an upward trend over time. This phenomenon has become a significant global health concern, leading to over 600 million cases annually and associated with a mortality rate of up to 420,000 premature deaths each year. The consumption of food that is contaminated with harmful bacteria, viruses, parasites, or chemical substances can result in the onset of various diseases [3]. It has been acknowledged that packaging, storage conditions, and transportation factors have a significant impact on food quality, safety, and shelf life. One promising solution that has been identified as a means of preventing food contamination is the use of active and intelligent packaging systems [4]. Active food packaging technology encompasses systems that incorporate active substances into the polymeric matrix. The embedded components exhibit antimicrobial, antifungal, antioxidant properties and are able to absorb or reduce oxygen, carbon dioxide or ethylene, thereby enhancing the quality and safety of food products [5].

The classification of nanomaterials is based on their structural features and functional properties. Specifically, nanomaterials can be categorized into the following groups: zero-dimensional (0D) nanoparticles (such as metal and metal oxide nanoparticles, quantum dots, silica (SiO₂) nanoparticles); one-dimensional (1D) nanotubes (CNTs), metal nanowires or nanofibers; two-dimensional (2D) nanosheets (graphene and derivatives, carbon nitride nanosheets and layered double hydroxide); and three-dimensional (3D) structures (metal–organic framework, covalent organic framework or nanocomposites) [6].

In the extensive array of nanomaterials, two-dimensional (2D) materials have emerged as a particularly promising class. These materials have demonstrated considerable potential



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in the domain of active and intelligent food packaging due to their properties, such as a large surface area, high mechanical performance, high barrier, easy functionalization and high electrical conductivity [7]. The embedded components exhibit antimicrobial, antifungal, and antioxidant properties and are able to absorb or reduce oxygen, carbon dioxide or ethylene, thereby enhancing the quality and safety of food products [5].

The utilization of two-dimensional materials, such as graphene, has facilitated the advent of novel avenues for the advancement of active packaging (AP). The integration of these materials with polymers has the potential to enhance the barrier, thermal, mechanical and bioactive properties of packaging [8,9].

The objective of this paper is to provide a comprehensive overview of patented two-dimensional materials in the field of active packaging.

Patents function not only as legal documents but also as a valuable repository of technical information, which is often not readily available elsewhere.

The retrieval of this information necessitates the interrogation of patent databases, which serve as repositories for a substantial volume of invention data that are subject to continuous updating.

A plethora of patent databases are currently available for use, as evidenced by the list provided on the WIPO (World Intellectual Property Organization) website [10]. The decision to utilize Espacenet [11] as a primary database was motivated by its cost-free nature and its substantial document collection, which encompasses over 150 million records. The aforementioned database boasts a comprehensive global scope, enabling users to seek information concerning published patent applications from over 90 patent-granting authorities.

2. Materials and Methods

A series of searches were conducted using Espacenet, a database provided by the European Patent Office (EPO), to analyze patents reflecting the main trends in the use of 2D nanomaterials in active food packaging. A combination of keywords in the title/abstract/claims search fields with Boolean and proximity operators and classification codes was used in the searches.

The exhaustive list of search queries employed to retrieve patent data can be found in Appendix A. Given that Espacenet is a freely accessible database, any researcher is capable of reproducing and retrieving updated data by simply copying and pasting the query into the “Smart Search Editor” on Espacenet.

Classification symbols were retrieved by means of the Espacenet classification search tool [12], the WIPO IPCCAT-neural system [13] and the new CPC (Cooperative Patent Classification) text categorizer [14], powered by AI and provided by the EPO.

A summary of the IPC (International Patent Classification) and CPC (Cooperative Patent Classification) codes used in the search is shown in Table 1.

Table 1. List of classification codes used in the patent searches.

Classification Code	System	Definition
B65D+	IPC/CPC	Containers for storage or transport of articles or materials
B32B27+	IPC/CPC	Layered products comprising a layer of synthetic resin
C08J5/18	IPC/CPC	Manufacture of films or sheets
Y02A40/90	CPC	Adaptation technologies in food processing or handling: food conservation
A23V2002/00	CPC (Indexing scheme)	Food compositions, function of food ingredients or processes for food or foodstuffs
B32B2439/70	CPC	Food packaging

Table 1. Cont.

Classification Code	System	Definition
Y02W90/10	CPC	Bio-packaging
C01B32/182	IPC/CPC	Graphene
C08K3/042	CPC	Use of carbon as compounding ingredient—Graphene or derivatives
C01B32/198	IPC/CPC	Graphene oxide
C01B21/064	IPC/CPC	Binary compounds of nitrogen with boron
C08K3/38	IPC/CPC	Boron-containing compounds
C08K2003/385	CPC	Binary compounds of nitrogen with boron
C01F7/785	IPC/CPC	Hydrotalcite
C08K3/14	IPC/CPC	Carbides

3. Results and Discussion

The search was initiated with a basic query in the title/abstract/claims field to ascertain the classification of patents. The query was formulated as follows: `ctxt all "food packaging"`; it yielded 110,432 results.

The IPC symbols that were retrieved and identified as primary were A23L, B32B27, C08J5/18, and B65D+. The corresponding definitions can be found in Table 1.

A similar procedure was employed for the CPC symbols, which are listed below: A23V2002/00, Y02W90/10, Y02A40/90 and B32B2439/70 (the definitions are also contained in Table 1).

All these classification symbols were used to construct the primary search query, which is as follows:

`(ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")) [QUERY 1].`

This query yielded 30,857 results (database accessed on 31 December 2024).

The results were analyzed according to the earliest priority date of filing and within the time frame between 2003 and 2022, which contains 21,222 patent documents. As illustrated in Figure 1, there is a discernible upward trend, with a slope change between 2014 and 2016, followed by a period in which the trend remains constant.

A series of queries were developed in order to identify patent documents that claim 2D nanomaterials. These queries are reported in Table A1 (see Appendix A).

A review of the literature revealed that the most widely utilized 2D materials in active food packaging systems were graphene and its derivatives, including graphene oxide (GO) and reduced graphene oxide (rGO), as well as MXenes and silicate clay [7].

The patent analysis aligns closely with the scientific literature, indicating that the top five two-dimensional nanomaterials include montmorillonite, graphene, GO, LDHs (layered double hydroxides), and vermiculite (as illustrated in Figure 2).

An exhaustive examination of priority filings from 2018 to 2022 reveals that carbon-based nanomaterials and MXenes have been increasingly claimed in patent applications (see Figure 3).

As illustrated in Table 2, the primary methods of graphene preparation are exfoliation and chemical vapor deposition (CVD).

The patent data set did not include other recently developed carbon-based nanomaterials, including hydrogenated graphene (graphane), fluorinated graphene (fluorographene), and graphene introduced by acetylenic chains (graphyne and graphdiyne).

In addition to graphene derivatives, another family of 2D compounds, specifically transition metal carbides and/or nitrides, known as “MXenes”, has garnered attention in the field of food packaging due to their unique properties. While the number of patents related to MXenes is currently limited, the potential implications of this emerging material are significant.

$\text{Ti}_3\text{C}_2\text{T}_x$, a 2D MXene that has been extensively studied and claimed, is produced by etching the Al layers in Ti_3AlC_2 , followed by exfoliation.

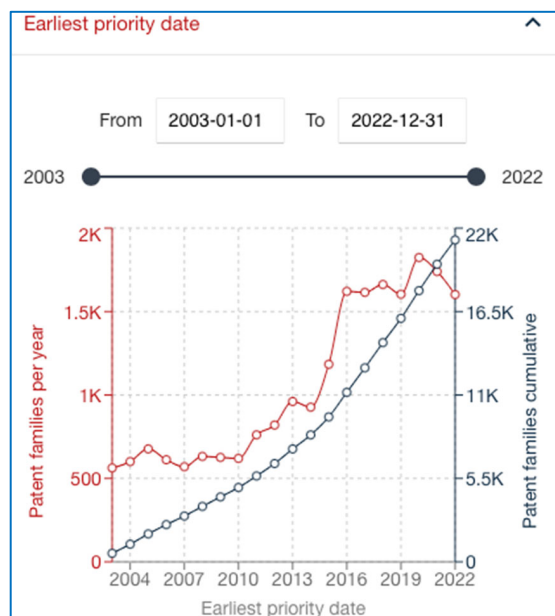


Figure 1. Trend of filings on the basis of the earliest priority date.

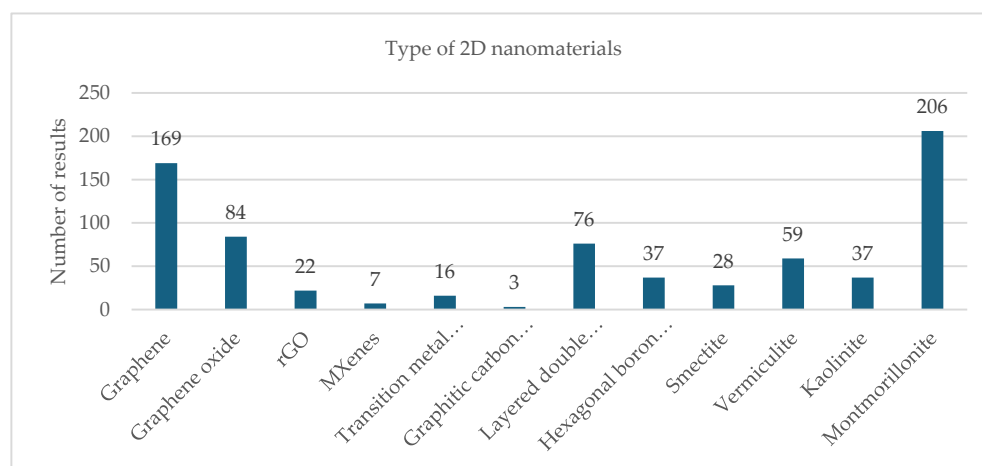


Figure 2. Type of 2D nanomaterials claimed in patent documents.

Table 2. Methods for preparing graphene.

Preparation of Graphene	Corresponding IPC/CPC Symbol	No. of Results
CVD	C01B32/186	14
Epitaxial growth	C01B32/188	1
Exfoliation	C01B32/19	15
Exfoliation—starting from graphitic oxides	C01B32/192	4

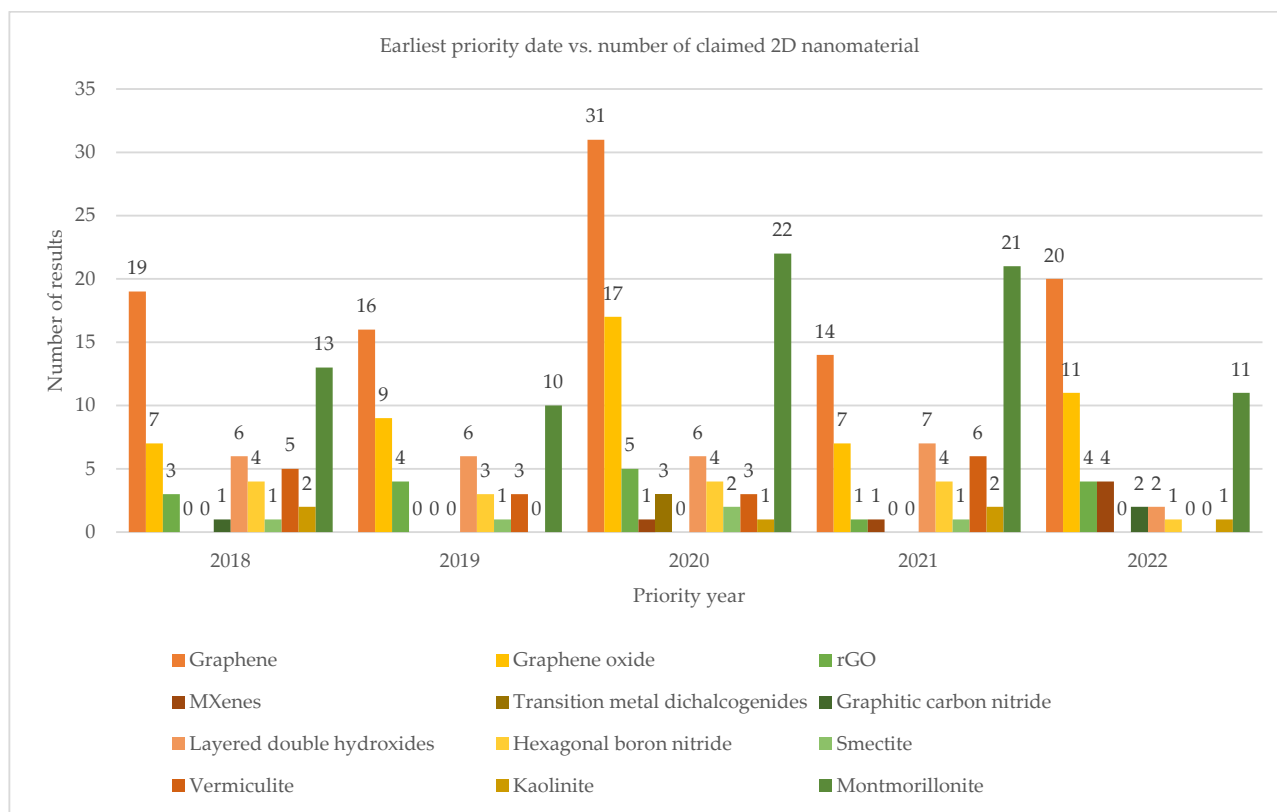


Figure 3. Distribution of 2D nanomaterials in patents from 2018 to 2022.

A compendium of additional compositions, as delineated within the scope of patent documents, is enumerated in Table 3.

Table 3. List of MXene composition.

Patent/Patent Application Number	MXene Formula
EP4349881A1 [15]	$M_{n+1}X_nT_x$, M is titanium and/or a combination of titanium and at least one of Sc, Y, Zr, Hf, V, Nb, Ta, Cr, Mo and W, X is carbon and/or nitrogen, n is any integral of 1–4, and T_x is -O, OH or -F.
CN114591606A [16]	$M_{n+1}X_nT_x$, wherein, M is a pre-transition metal element, and X is a carbon and/or nitrogen, n is any integer from 1 to 4, T_x is -O, -OH or -F
CN112662014A [17]	$Ti_3C_2T_x$
CN115558152A [18]	$Ti_3C_2T_x$
CN115418089A [19]	MXene nanosheets are titanium carbide nanosheets, titanium nitride nanosheets, titanium carbonitride nanosheets, zirconium carbide nanosheets, molybdenum carbide nanosheets, and vanadium carbide nanosheets
CN114560990A [20]	Ti_3C_2 , Nb_2C or Ti_3AlC_2
CN116531575A [21]	$Ti_3C_2T_x$, Ti_2CT_x , Nb_2CT_x , Mo_2CT_x , $Ti_4N_3T_x$, $Ta_4C_3T_x$, V_2CT_x , $Zr_3C_2T_x$, $(Nb_{0.8}Zr_{0.2})_4C_3T_x$

A compelling family of compounds that merits further consideration is that of LDHs, also named as anionic clays or hydrotalcite-like compounds, which are distinguished by their mechanical, barrier, and antimicrobial properties.

These substances are utilized as fillers in biopolymers, such as cellulose and polylactic acid (PLA) and polyvinyl alcohol (PVA). Additionally, they are frequently employed in

blends with metal nanoparticles, including zinc oxide and titanium dioxide, which serve to augment the antimicrobial properties of the nanocomposite.

A survey of the patent literature reveals that the most frequently utilized biopolymers in conjunction with two-dimensional nanomaterials are cellulose, polyvinyl alcohol, starch, polylactic acid, and chitosan [22] (see Figure 4).

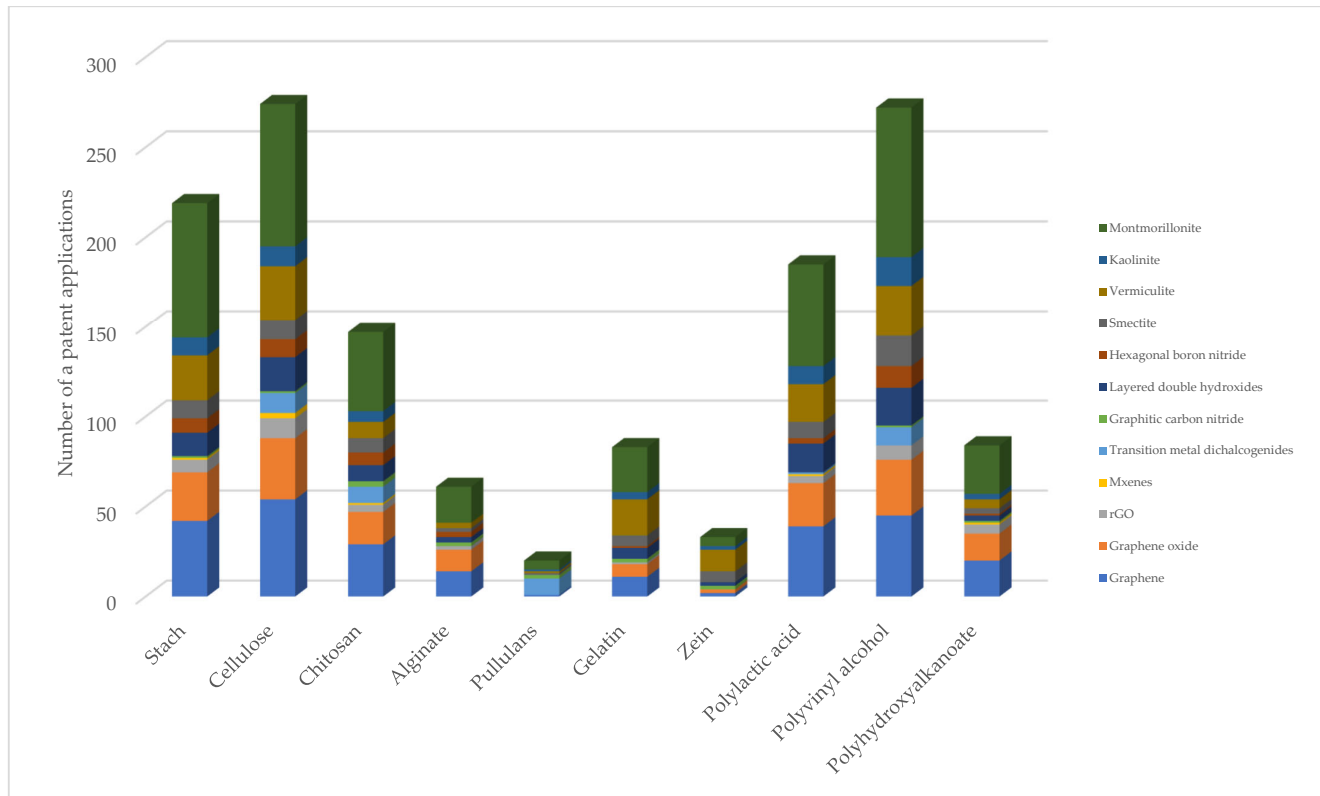


Figure 4. Biopolymer type vs. 2D nanomaterial.

From a legal perspective, as well as from a geographic point of view, China has been identified as the country with the highest number of patent applications filed for 2D materials in AP technology. The United States and Japan have been identified as the countries with the third- and fourth-highest numbers of patent applications, respectively, after the international applications.

A significant number of applicants have opted to utilize the PCT (Patent Cooperation Treaty) procedure, which permits the postponement of entry into the national phases (see Figure 5). International applications offer applicants the opportunity to seek provisional protection in multiple countries through a single application. The PCT currently has 158 contracting states. At the conclusion of the 30/32-month period following the priority filing, a PCT application expires, and the applicant must determine whether it is advantageous (from an economic or a strategic perspective) to enter national phases (e.g., China, USA, Europe, etc.).

This procedural decision is made when the applicant lacks knowledge regarding the specific states in which commercialization of the invention is desired, or when the invention itself possesses a low TRL (Technology Readiness Level).

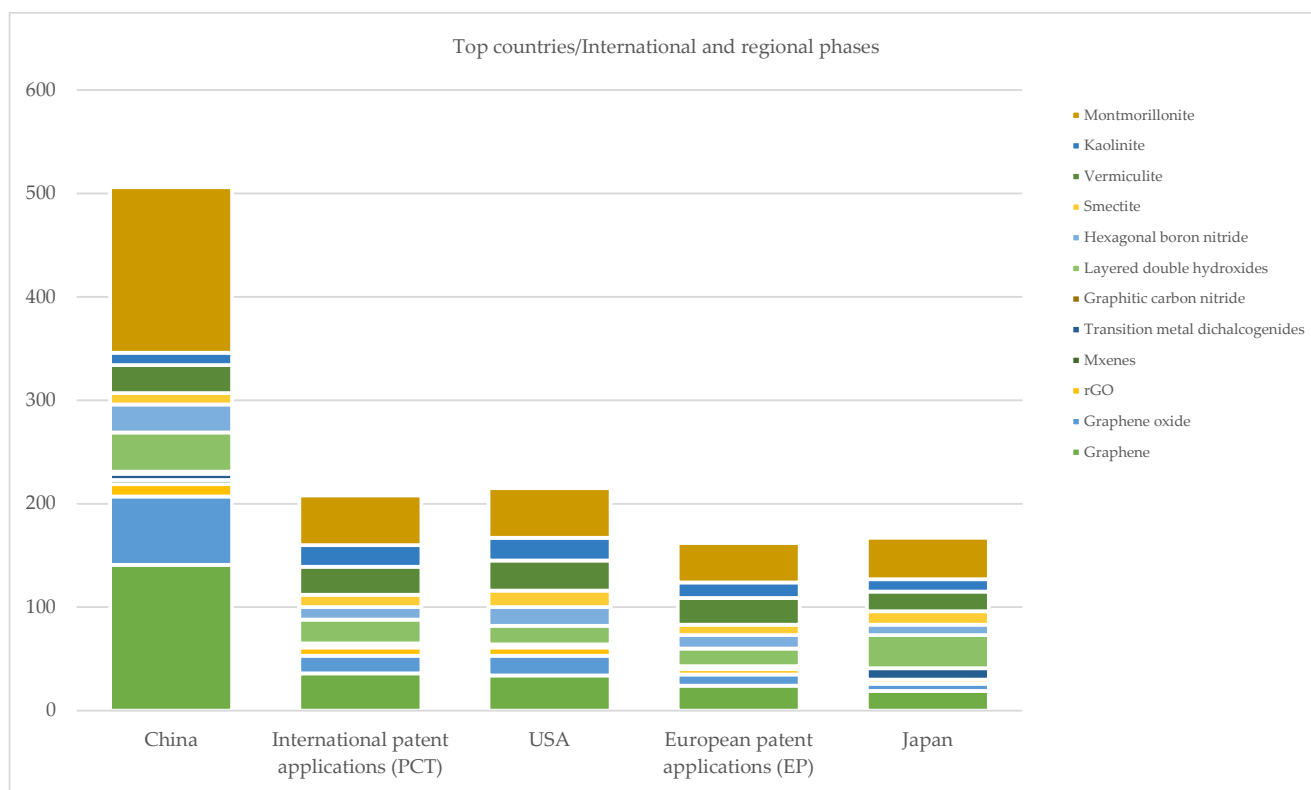


Figure 5. Top countries per number of filed patent/patent applications.

4. Conclusions

China, the USA and Japan are the countries with the highest number of filings. In addition, the PCT procedure is utilized extensively by applicants, occupying the third position in terms of the number of filed applications.

In terms of the materials most frequently claimed in patent applications, carbon allotropes are the most prevalent, together with silicate-clay-based 2D materials.

Montmorillonite, graphene and graphene oxide are the most frequently claimed compounds in patents, followed by vermiculite, kaolinite, layered double hydroxides and hexagonal boron nitride.

Other two-dimensional materials, such as transition metal dichalcogenides, reduced graphene oxide, MXenes and graphitic carbon nitrides ($g-C_3N_4$), have been the subject of fewer patent applications.

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Conflicts of Interest: The author declares no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

GO	Graphene oxide
rGO	Reduced graphene oxide
$g-C_3N_4$	Graphitic carbon nitrides
h-BN	Hexagonal boron nitride

LDHs	Layered double hydroxides
TDMs	Transition metal dichalcogenides
PLA	Polylactic acid
PVA	Polyvinyl alcohol
PHA	Polyhydroxyalkanoate
EPO	European Patent Office
WIPO	World Intellectual Property Organization

Appendix A

This appendix provides a list of the search queries that were used to retrieve patent data.

Table A1. List of queries for retrieval of 2D nanomaterials in patents.

2D Nanomaterial	Search Query
Hexagonal boron nitride (h-BN)	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")))) AND (ctxt all "hexagonal boron nitride?" OR cl all "C01B21/064" OR cl all "C08K3/38" OR cpc all "C08K2003/385")
Layered double hydroxides (LDHs)	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")))) AND (ctxt all "layered double hydroxide?" OR cl all "C01F7/785" OR ctxt all "anionic clay?" OR ctxt all "hydrotalcite")
Graphitic carbon nitride (g-C ₃ N ₄)	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")))) AND (ftxt all "graphitic carbon nitride?" OR ctxt all "g-C3N4")
Transition metal dichalcogenides (TDMs)	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")))) AND (ftxt all "Transition metal dichalcogenide?" OR ctxt all "molybdenum disulfide" OR ctxt all "molybdenum diselenide" OR ctxt all "molybdenum ditelluride")
Graphene	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")))) AND (ctxt all "graphene" OR cl =/low "C01B32/182" OR cl =/low "C08K3/042")
Graphene oxide (GO)	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")))) AND (ctxt = ("graphene "prox/ordered "oxide") OR cl =/low "C01B32/198")
Reduced graphene oxide (rGO)	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")))) AND (ctxt = ("reduced graphene "prox/ordered "oxide") OR ctxt all "rGO")
MXenes	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")))) AND (ctxt all "MXene?" OR cl all "C08K3/14")
Smectite	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")))) AND ctxt all "smectite"

Table A1. Cont.

2D Nanomaterial	Search Query
Vermiculite	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")) AND ctxt all "vermiculite")
Kaolinite	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")) AND ctxt all "kaolinite")
Montmorillonite	((ctxt = ("food "prox/distance < 2 "packag*") AND (cl all "B65D" OR cl all "B32B27" OR cl all "C08J5/18" OR cl all "A23L")) OR cpc all "B32B2439/70" OR ((cpc all "Y02W90/10" OR cpc all "Y02A40/90" OR cpc all "A23V2002/00") AND ctxt = ("food "prox/distance < 2 "packag*")) AND ctxt all "montmorillonite")

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