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DOMENIUL FUNDAMENTAL – ȘTIINȚE INGINEREȘTI
DOMENIUL DE DOCTORAT – INGINERIE CHIMICĂ

TEZĂ DE DOCTORAT
Rezumat

Studiu privind sinteza sistemelor fotocatalitice de tipul materialelor nanocompozite hibride semiconductoare utilizate pentru diferite aplicații

Autor: Usman Abubakar Katsina

Conducător științific: Prof. dr. ing. habil. Cursaru Diana-Luciana

Ploiești 2024

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The synthesis of photocatalytic systems based on semiconductor hybrid nanocomposite materials for various applications.

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CUPRINS

REZUMAT

Procesul de industrializare și urbanizare accelerată a creat provocări considerabile pentru durabilitatea energetică și conservarea mediului, ducând la căutarea de soluții alternative la nivel global. Fotocataliza are un mare potențial pentru rezolvarea acestei probleme, deoarece poate transforma cu ușurință energia solară în combustibili chimici și poate facilita remedierea mediului prin valorificarea catalizatorilor semiconductori. Eficacitatea fotocatalizei semiconductoare depinde în mare măsură de designul și caracteristicile sale, urmărind să absoarbă lumina întregului spectru solar și să genereze purtători de sarcină activi capabili să conducă reacții fotocatalitice. Nitrura de carbon grafitică polimerică (g-C_3N_4) a fost considerată un fotocatalizator semiconductor promițător datorită caracteristicilor sale unice, inclusiv flexibilitatea mecanică, mobilitatea ridicată a purtătorului de sarcină și stabilitatea ridicată. Este abundent, ușor de fabricat și a prezentat o eficiență excelentă în fotodegradarea contaminanților nocivi în soluții apoase. Cu toate acestea, viabilitatea comercială a g-C_3N_4 și a altor semiconductori unici este împiedicată de intervalul lor îngust de absorbție a luminii, recombinarea rapidă a purtătorilor de sarcină, eficiența cuantică slabă și potențialul redox limitat. Se poate spune că cele mai semnificative progrese în prezent se învârt în jurul dopajului nemetalic al nitrurii de carbon (CN) și al creării de fotocatalizatori heterostructurați pe bază de CN care prezintă o eficacitate fotocatalitică și o fotostabilitate excepționale. Această teză s-a concentrat pe fabricarea și compararea compozitelor heterostructurate de g-C_3N_4 (CN) dopate cu oxigen și fosfor cu oxizi metalici binari de α-Bi_2O_3 (α-BO), α-Fe_2O_3 (α-FO) și perovskit $BiFeO_3$ (BFO), cu aplicație potențială în degradarea fotocatalitică a colorantului apos de rodamină B (RhB). Structurile CN concepute electronic au fost inițial obținute printr-o polimerizare termică a ureei. Semiconductorul CN pur a fost apoi dopat individual cu atomi de oxigen (O) și fosfor (P) pentru a ajusta caracteristicile originale ale CN, formând OCN și PCN. CN dopat cu O a fost sintetizat folosind 5, 10, 20 și 30% gr. apă oxigenată (H_2O_2) ca precursor bogat în O, obținându-se OCN-1, OCN-2, OCN-3 și respectiv, OCN-4. Dopajul cu P al semiconductorilor CN a fost realizat fie prin policondensarea într-o singură etapă a amestecului de uree și hipofosfit de sodiu monohidrat ($NaPO_2H_2 \cdot H_2O$) pentru a obține PCN, fie prin polimerizarea termică a amestecului de CN pur și $NaPO_2H_2 \cdot H_2O$ pentru a obține PCNE. În fiecare amestec s-a utilizat 10 și 15% gr. din precursorul $NaPO_2H_2 \cdot H_2O$, obținându-se PCN-1, PCNE-1, PCN-2 și, respectiv PCNE-2. Fiecare dintre α-BO, α-FO și BFO a fost sintetizat hidrotermal și a format un compozit heterostructurat cu materialele OCN și PCN printr-o cale chimică simplă

în prezență de metanol. Efectul morfologiei BFO asupra arhitecturii structurale și a activității fotocatalitice a compozitelor au fost, de asemenea, evaluate prin sintetizarea BFO folosind metoda hidrotermală asistată de uree (U-BFO) și metoda sol-gel (SG-BFO). Au fost utilizate diferite tehnici de caracterizare analitică pentru a investiga cristalinitatea fazei, compoziția chimică, proprietățile suprafeței și comportamentele opto-electronice ale probelor sintetizate. Analiza probelor a indicat că încorporarea de α-BO, α-FO și BFO, împreună cu doparea cu oxigen și fosfor, a îmbunătățit semnificativ structura porilor, a lărgit intervalul de absorbție a luminii vizibile și a îmbunătățit eficiența separării purtătorilor de sarcină în nitrură de carbon pură. Aceste efecte sinergice au oferit mai mulți centri activi pentru adsorbția de O_2 și H^+, au promovat generarea de electroni și au inhibat recombinarea e/h+, sporind astfel activitatea de fotodegradare. Impactul dopajului a fost elucidat în degradarea fotocatalitică a RhB, prezentând eficiențe de 88,5% și 95,6% pentru OCN-1 și respectiv, PCNE-2. Interacțiunile sinergice dintre α-BO, α-FO și BFO cu CN dopat cu O și P au îmbunătățit și mai mult eficacitatea fotocatalitică a catalizatorilor, compozitele suportate de PCN demonstrând o eficacitate fotocatalitică superioară comparativ cu compozitele pe bază de OCN, în ciuda faptului că acestea din urmă au o suprafață relativ mai mare. Acest lucru sugerează că eficiența fotodegradării a fost afectată de factori care se extind dincolo de simplele proprietăți de adsorbție ale catalizatorilor. Posibilii parametri care contribuie la performanța îmbunătățită a compozitelor pe bază de PCN au fost descoperiți prin investigațiile UV-VIS și PL, arătând o separare îmbunătățită a sarcinii, proprietăți electronice mai favorabile și o absorbție îmbunătățită a luminii, facilitând fotoreacții mai eficiente. În plus, mediul chimic furnizat de dopajul cu fosfor ar putea crea interacțiuni benefice care conduc la o activitate catalitică mai mare. Cea mai bună eficiență de degradare de 99,2% a fost prezentată de heterostructurile cu schema Z U-BFO/PCN în 20 de minute de iradiere cu lumină vizibilă, cu o cinetică de degradare remarcabilă de ordinul pseudo-întâi de 0,253 min^{-1}. S-a demonstrat că radicalii superoxid ($\bullet O_2^-$) și radicalii hidroxil ($\bullet OH^-$) joacă un rol major în îndepărtarea RhB folosind heterojoncțiunile U-BFO/OCN, U-BFO/PCN, α-BO/PCN și α-FO/PCN printr-un mecanism de transfer de tip schema Z. Astfel, se ajunge la concluzia că sistemele de heterojoncțiune cu schema Z nemetalice bazate pe g-C_3N_4 depășesc semnificativ alte materiale semiconductoare în degradarea RhB.

Această teză intitulată “**Studiu privind sinteza sistemelor fotocatalitice de tipul materialelor nanocompozite hibride semiconductoare utilizate pentru diferite aplicații**”, este structurată în șase capitole după cum urmează:

Capitolul I

Primul capitol, "**Introducere generală**” acoperă informații generale despre fotocataliza eterogenă a semiconductoarelor și aplicarea acesteia în soluțiile de protecție a mediului și de energie durabilă. De asemenea, cuprinde enunțul problemei cercetării, scopurile, obiectivele și semnificația cercetării. Capitolul I a trecut în revistă problemele majore provocate de industrializarea și urbanizarea globală, care necesită luarea în considerare imediată și promptă cu privire la sustenabilitatea energetică și a mediului. De asemenea, a subliniat faptul că semnificația cercetării transcende investigațiile academice și oferă un impact pozitiv în domeniile științei materialelor și ingineriei durabile. În plus, oferă soluții durabile și strategii pentru înțelegerea fotocatalizatorilor avansați sintetizați pentru tratarea apelor uzate, în paralel cu imperativul global de a asigura calitatea apei și a practicilor durabile în fața intensificării provocărilor legate de mediu.

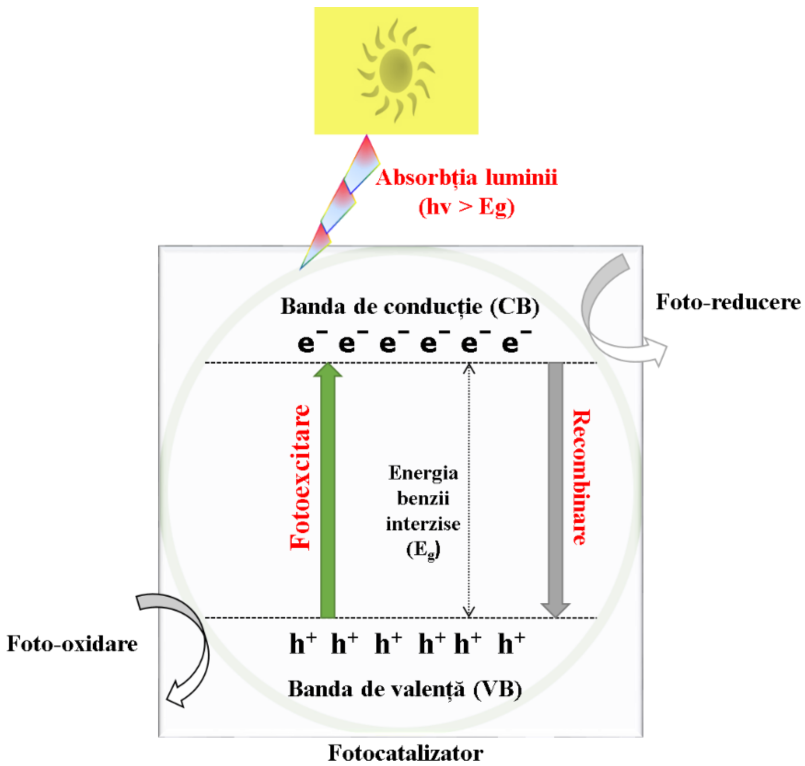

Figura 1: Ilustrare schematică a mecanismului general pentru sistemul fotocatalitic.

Capitolul II

Al doilea capitol, „**Date de literatură**” prezintă și trece în revistă ultimele cercetării privind materialele pe bază de g-C_3N_4 în aplicații fotocatalitice, subliniind degradarea poluanților și succesele remarcabile în modificarea și formarea heterojoncțiunilor acestora.

Sinteza g-C_3N_4 implică precursori bogați în azot precum ureea și melamina, rezultând structuri stabile bazate pe tri-s-triazină în condiții controlate. Tehnici precum termoliza la temperaturi înalte și metodele cu template ajustează proprietățile morfologice și optice ale g-C_3N_4, esențiale pentru îmbunătățirea eficienței sale fotocatalitice. În plus, doparea elementală și moleculară ajustează banda interzisă a g-C_3N_4, extinzând capacitățile de absorbție a luminii și îmbunătățind separarea sarcinilor. Nanotehnologia joacă un rol crucial în optimizarea caracteristicilor de suprafață și transportul purtătorilor de sarcină ale g-C_3N_4. Diverse nanostructuri, inclusiv nanoparticule și foi nano, amplifică activitatea sa fotocatalitică prin maximizarea suprafeței și integrității structurale. Încărcarea cu cocatalizatori îmbunătățește și mai mult eficiența prin îmbunătățirea separării electron-gol și a chimiei interfețelor. Integrarea g-C_3N_4 în sisteme de heterojoncțiuni reprezintă un avans semnificativ, abordând limitările inerente și îmbunătățind performanța generală. Tipuri de heterojoncțiuni precum Tip-I, Tip-II, joncțiuni Schottky, izotip, anisotip și configurații de tip Z-scheme îmbunătățesc eficiența separării sarcinilor prin aliniamente de bandă optimizate și inginerie interfețială. Aceste sisteme demonstrează activități fotocatalitice superioare în diverse aplicații, cum ar fi descompunerea apei și degradarea poluanților. În plus, capitolul explorează diverse heterostructuri bazate pe g-C_3N_4, inclusiv oxizi metalici, oxizi metalici mixți, sulfuri metalice, cadre metal-organice (MOF) și sisteme g-C_3N_4 auto-infuzate. Fiecare tip de heterostructură valorifică proprietăți unice pentru a obține eficiențe fotocatalitice ridicate, exemplificate prin performanțele lor în degradarea poluanților organici și facilitarea producției de hidrogen. Următoarele figuri reprezintă diferitele tipuri de heterojoncțiuni de tip Z-scheme.

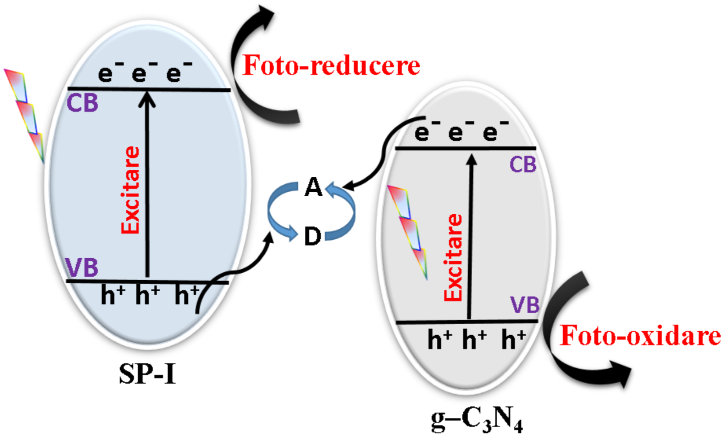

Figura 2: Ilustrare schematică a heterojoncțiunii tradiționale cu schema Z pentru g-C_3N_4.

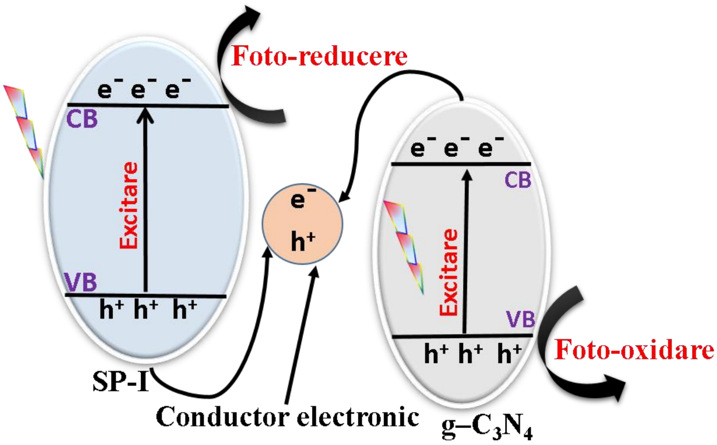

Figura 3: Ilustrare schematică a heterojoncțiunii cu schema Z în stare solidă bazată pe g-C_3N_4.

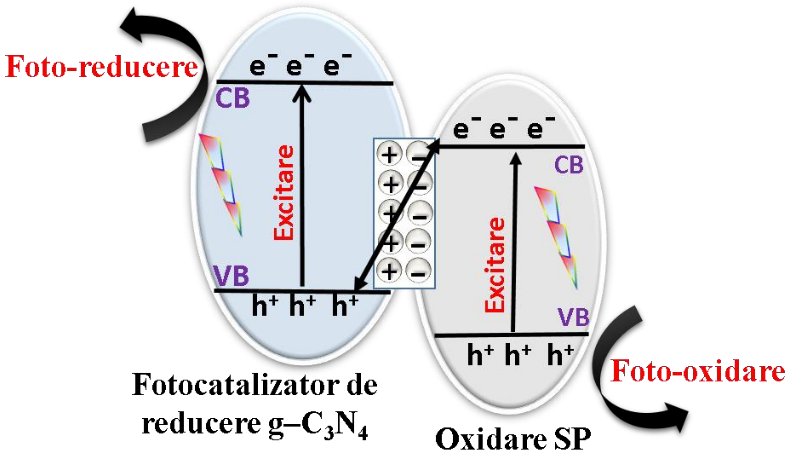

Figura 4: Ilustrare schematică a heterojoncțiunii cu schema Z directă g-C_3N_4

Capitolul III

Al treilea capitol, „**Materiale şi studiu experimental**” prezintă materialele, reactivii şi echipamentele utilizate în această cercetare, precum şi toate metodele de preparare şi caracterizare, precum şi experimentele de fotodegradare ale materialelor sintetizate. Capitolul III se concentrează pe materialele, metodologiile şi tehnicile de caracterizare utilizate în acest studiu, descriind toate metodele de sinteză conturate pentru fiecare catalizator (materialele pure şi heterojoncțiuni). Secțiunea de caracterizare analitică a materialelor prezintă câteva detalii referitoare la tehnicile şi echipamentele utilizate, incluzând microscopia electronică de baleiaj, spectroscopie cu dispersie de energie, spectroscopie în infraroşu cu transformată Fourier, difracție de raze X, spectroscopie de reflexie difuză ultraviolet-vizibil, fizisorbție, analiza termogravimetrică şi analiza termogravimetrică derivată. Capitolul se încheie cu o descriere a configurației programului experimental şi a procedurii de fotodegradare a Rodaminei B.

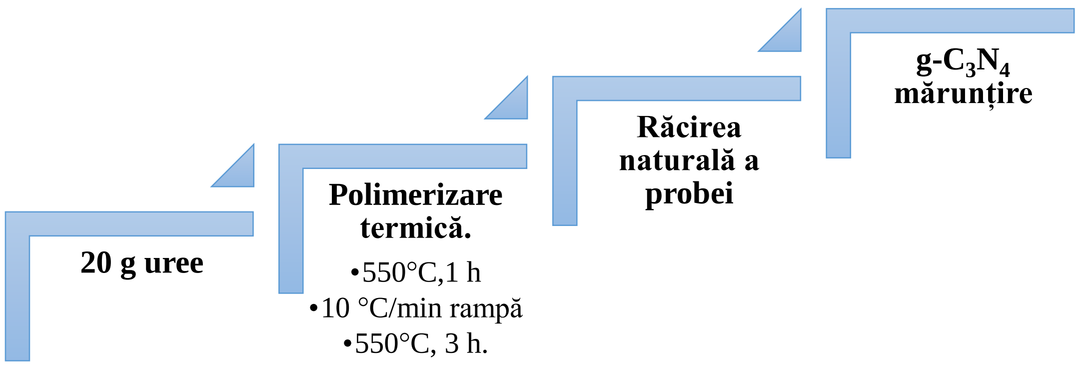

Figura 5: Diagrama folosită pentru sinteza g-C_3N_4 polimeric

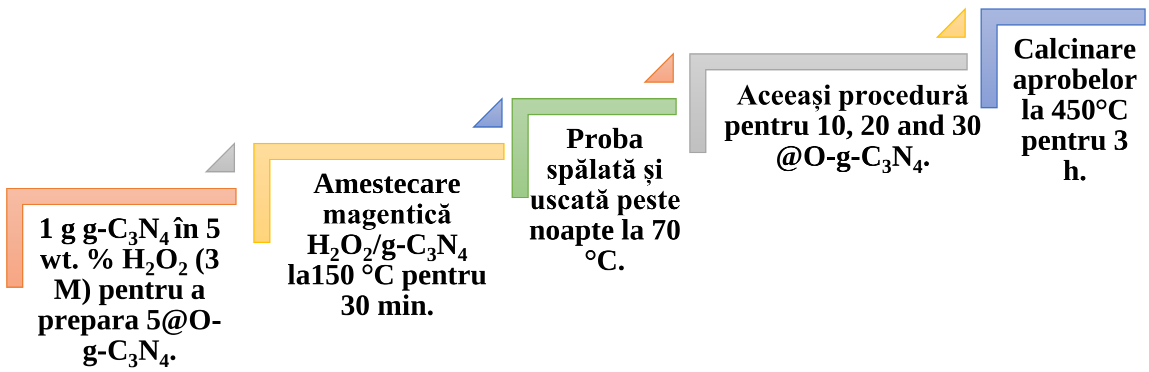

Figura 6: Diagrama sintezei g-C_3N_4 polimeric dopat cu oxigen

Capitolul IV

Al patrulea capitol, „**Caracterizarea materialelor multimodale**” prezintă și discută arhitectura structurală, compoziția chimică, caracteristicile suprafeței și proprietățile optoelectronice ale materialelor pure și compozite în trei secțiuni. Prima secțiune elucidează efectele dopajului, în timp ce a doua și a treia examinează construcția sistemelor de heterojoncțiune suportate pe g-C_3N_4 dopat cu catalizatori oxidici de Bi-Fe.Caracterizarea exhaustivă a furnizat informații valoroase cu privire la structura compozitelor, morfologia și proprietățile optice. Spectroscopia în infraroșu cu transformata Fourier (FT-IR) a furnizat informații despre structura chimică și grupările funcționale, indicând modificările suprafeței și posibilele interacțiuni. Difracția de raze X (DRX) a furnizat informaţii despre compozițiile fazelor, confirmând prezența fazelor cristaline pure în compozite. Studiul de adsorbţie fizică a completat informațiile privind suprafața, distribuția, dimensiunile porilor și porozitatea sistemelor compozite și heterojoncțiilor sintetizate, care au fost esențiale pentru adsorbția și proprietățile lor catalitice. Microscopia electronică de baleaj (SEM) şi spectroscopia EDS au permis vizualizarea morfologică și cartografierea elementară a probelor. Analiza termogravimetrică (TGA) şi Termogravimetria derivată (DTG) au oferit informații privind stabilitatea termică a probelor. Analiza UV-Vis a permis caracterizarea proprietăților optice, cum ar fi energia benzii interzise și absorbția, care sunt vitale pentru activitățile fotocatalitice și fotovoltaice. Studiile de fotoluminiscență (PL) au analizat recombinarea purtătorului de sarcină foto-excitat, oferind informații valoroase despre durata de viață și eficiența purtătorului de sarcină. Aceste tehnici au oferit perspective asupra proprietăților care guvernează performanța superioară a degradării RhB a anumitor heterojoncțiuni și de ce sistemele heterojoncționale excelează în comparație cu catalizatorul de bază. Infuzia de oxigen și fosfor a îmbunătățit catalizatorii g-C_3N_4, demonstrat prin analize de fizisorbție cu N_2, cu O-g-C_3N_4 prezentând o suprafață BET superioară și P-g-C_3N_4 având comportamente optoelectronice îmbunătățite. **Tabelul 1** prezintă suprafețele BET, dimensiunile porilor BJH și volumele porilor pentru toți catalizatorii. Compozitele U-BFO/PCN demonstrează prezența macroporilor la 61 nm în plus față de mezopori la 4 nm. **Tabelul 2** prezintă dimensiunile cristalitelor pentru probele pure și compozite.

Tabelul 1: Suprafața BET, diametrul mediu al porilor BJH și volumul porilor pentru probele de BFO și compozitelor dopate pe suport de CN

Catalizator	Aria suprafeței BET ($cm^2\ g^{-1}$)	Distribuția mărimii porilor BJH	
		Diametru mediu al porilor (nm)	**Volum total de pori ($cm^3\ g^{-1}$)**
OCN	59,506	3,943	0,607
PCN	46,557	34,497	0,383
BFO/OCN	47,712	3,943	0,281
BFO/PCN	47,246	2,661	0,365
U-BFO/OCN	85,765	3,940	0,611
U-BFO/PCN	57,188	61,844	0,645
SG-BFO/OCN	41,587	3,936	0,331
SG-BFO/PCN	39,862	3,933	0,260
BFO	15,615	2,127	0,109
U-BFO	6,127	2,659	0,094
SG-BFO	27,584	3,930	0,142

Tabelul 2: Dimensiunile cristalelor pentru probele pure și compozite obținute din valorile FWHM pentru picurile de difracţie reprezentative.

Catalizator	Dimensiunea cristalitelor (nm)
BFO	10,64
U-BFO	22,99
SG-BFO	12,26
BFO/OCN	9,72
BFO/PCN	9,94
U-BFO/OCN	18,59
U-BFO/PCN	19,01
SG-BFO/OCN	7,36
SG-BFO/PCN	7,49

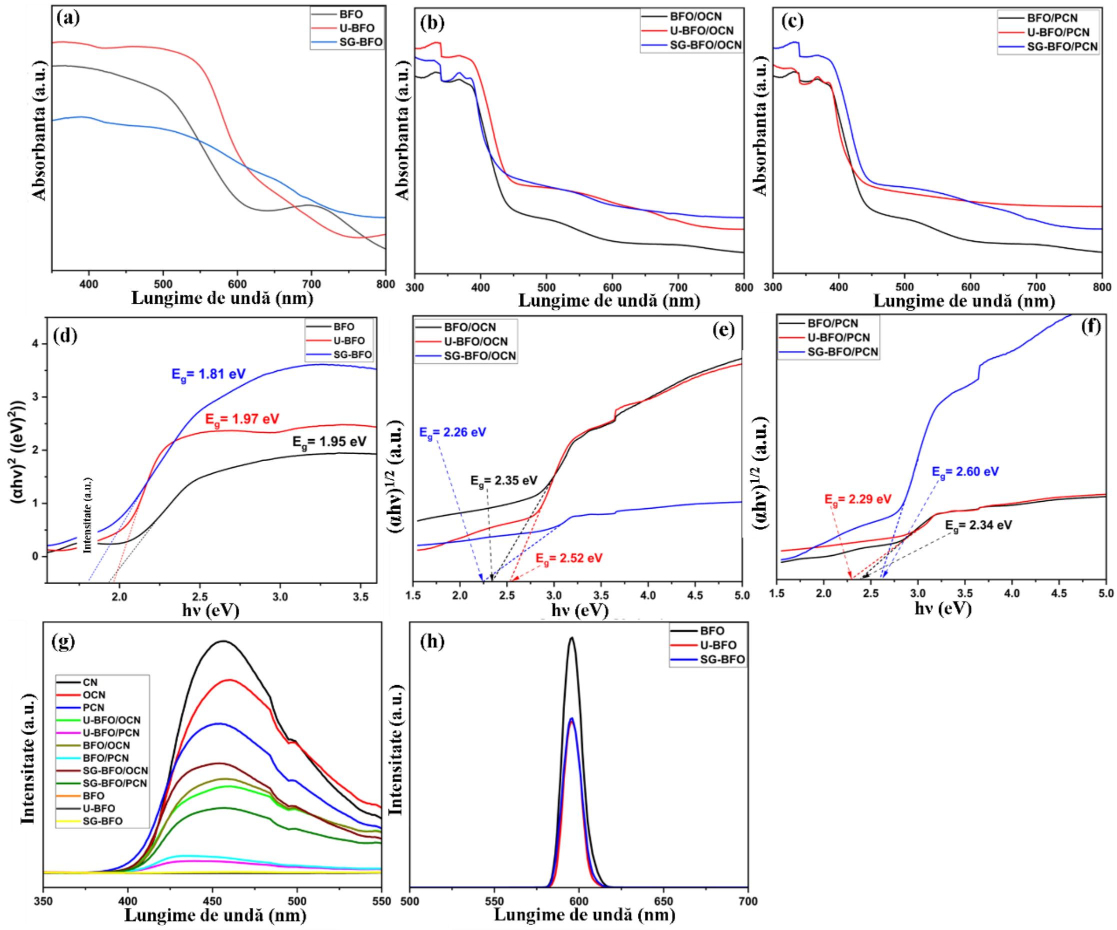

Figura 7: Spectrele de absorbție UV-VIS (a, b și c) și graficele Tauc corespunzătoare (d, e și f) pentru diferite probe de ferită de bismut, compozite dopate cu O și P; Spectrele PL pentru toate probele (g) și cu ferită de bismut (h)

Capitolul V

Al cincilea capitol „**Investigații de degradare fotocatalitică**" evaluează efectele diferitelor condiții experimentale asupra activității fotocatalitice și cineticii fotodegradării, propune mecanisme plauzibile de degradare și elucidează fotostabilitatea și fezabilitatea economică a sistemelor de heterojoncțiune. Catalizatorul P-g-C_3N_4 a obținut o eficiență de mineralizare a RhB de 95,6% și o viteză de degradare de 0,0672 min^{-1} în 30 de minute. Compozitele heterojoncționale cu α-Bi_2O_3 și α-Fe_2O_3 integrate în O-g-C_3N_4 și P-g-C_3N_4 au fost studiate pentru degradarea RhB sub lumină vizibilă, iar P-g- g-C_3N_4 cu α-Bi_2O_3 și α-Fe_2O_3 a arătat caracteristici optoelectronice superioare și o viteză de degradare mai bună decât catalizatorii puri. Configurațiile morfologice diferite ale $BiFeO_3$ în structurile heterojoncționale au fost, de asemenea, investigate, iar compozitul $BiFeO_3$/P-g-C_3N_4 cu formă eliptică a demonstrat cea mai bună eficiență de degradare a RhB de 99,2% și o constantă a vitezei reacției de 0,253 min^{-1}, urmând un mecanism de transfer al schemei Z cu radicali superoxid ($^{\bullet}O_2^-$) și ($^{\bullet}OH^-$) ca specii active principale. Compozitele au menținut o eficiență de 97% după patru cicluri de degradare, demonstrând fotostabilitate și fezabilitate economică impresionantă. Toate sistemele de heterojoncțiuni au urmat un mecanism de transfer de tip schemă Z în degradarea RhB, așa cum este prezentat mai jos. Compozitele heterojoncționale astfel sintetizate au prezentat, de asemenea, o fotostabilitate excelentă.

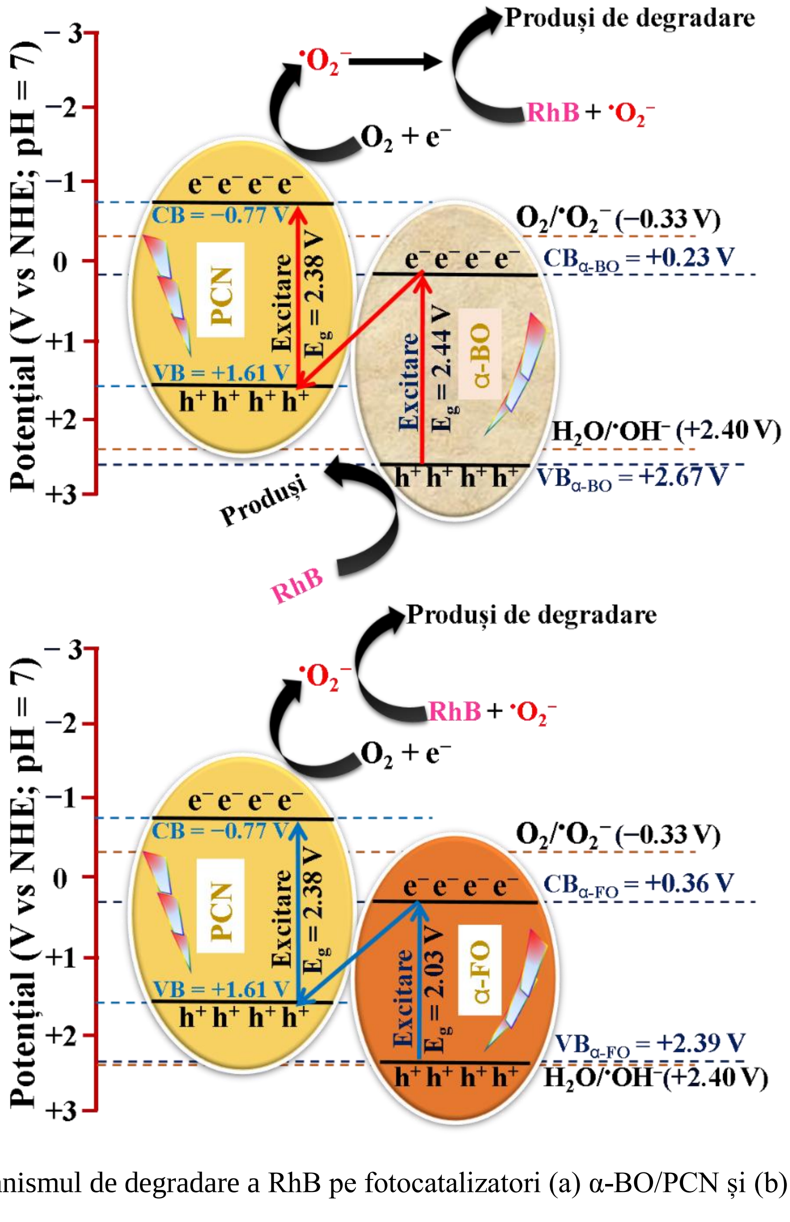

Figura 8: Mecanismul de degradare a RhB pe fotocatalizatori (a) α-BO/PCN și (b) α-FO/PCN.

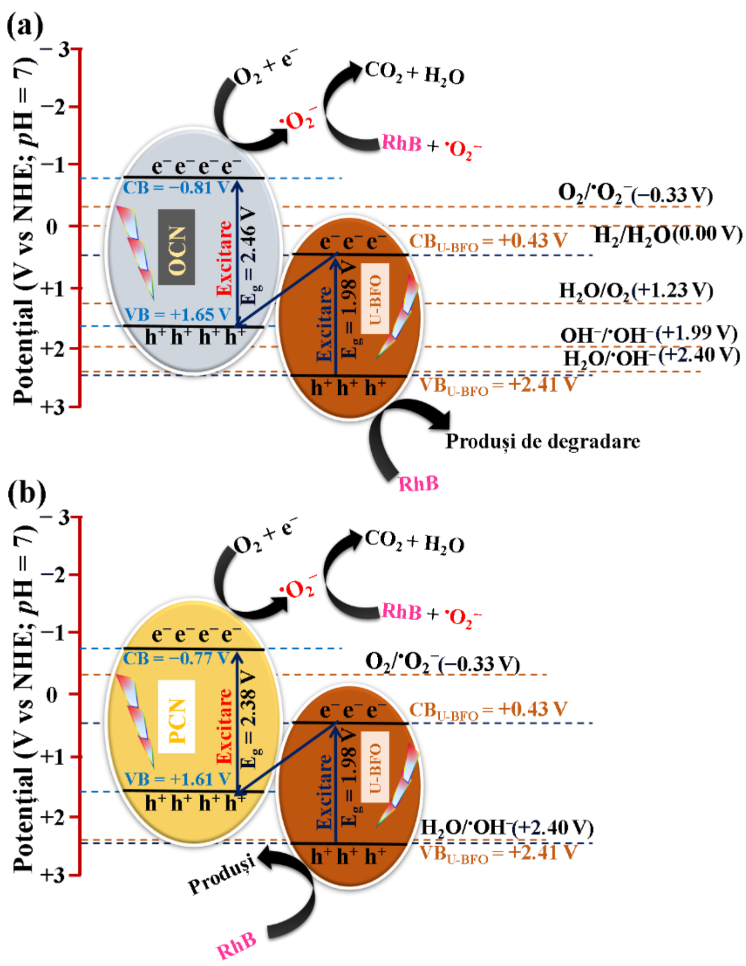

Figura 9: Mecanismul de degradare a RhB pentru fotocatalizatori (a) U-BFO/OCN și (b) U-BFO/PCN.

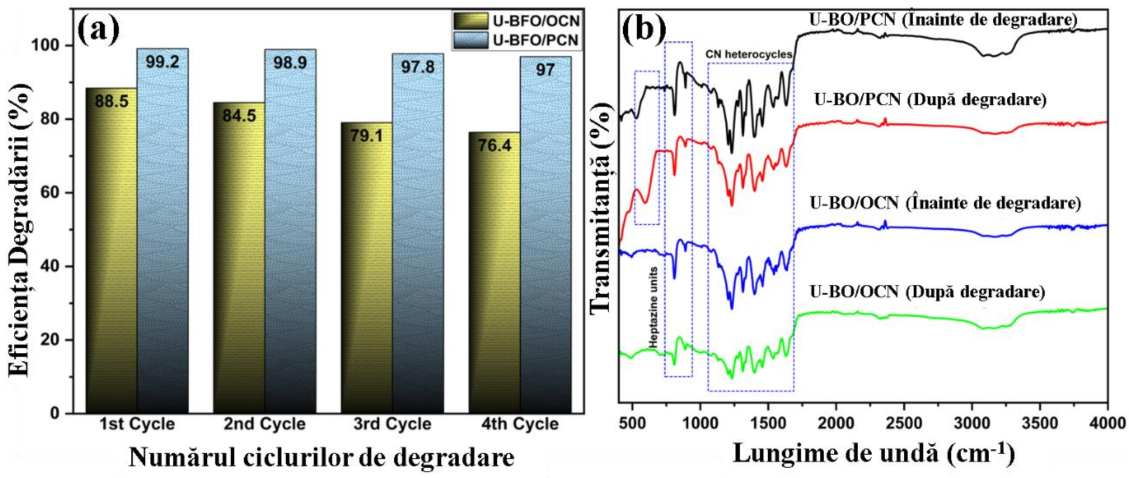

Figura 10: Test de reciclabilitate pentru fotocatalizatorii U-BFO/OCN și U-BFO/PCN.

Tabelul 3 prezintă diferite compozite suportate pe g-C_3N_4 cu mai mulți oxizi metalici amestecați pentru comparație cu compozitele de heterojoncțiune U-BFO/OCN și U-BFO/PCN.

Tabelul 3. Comparația unor fotocatalizatori de oxid de metal mixt pe suport de g-C_3N_4 pentru fotodegradarea coloranților în medii apoase

Catalizator (cantitate)	**Condiții**	**Eficiență (%) – constanta de viteză**	**Referințe**
$BaTiO_3$/g-C_3N_4 (25 mg)	50 mL (Albastru de metil, 10 mg/L) – Iradiere solară	91 %	(Kappadan și colab., 2021)
$CaTiO_3$/g-C_3N_4 (4 mg)	10 mL (Albastru de metil, 5 mg/L) –Lampă cu Tungsten 40 W	78 % - 0,046 min^{-1}	(Foroughipour și Nezamzadeh-Ejhieh, 2023)
$CoMoO_4$@g-C_3N_4 (10 mg)	50 mL (RhB, 20 mg/L) –Iradiere UV 300 W Xe	92 %	(Rabani și colab., 2021)
$ZnFe_2O_4$/g-C_3N_4 (0.5 g)	150 mL (RhB, 150 mg/L) –Lumină vizibilă 500 W Xe	98 % - 0,1955 min^{-1}	(Renukadevi și Pricilla Jeyakumari, 2020)
$BiFeO_3$/g-C_3N_4 (50 mg)	50 mL (Roșu Alzerid S) – Lumină vizibilă 300 W Xe	52 %	(Zaman și colab., 2023)
$MgAl_2O_4$/g-C_3N_4 (0.9 g)	100 mL (Roșu de metil 195, 60 ppm) – Lumină UV-vis 300 W Xe	100 % - 0,0663 min^{-1}	(Zehtab Salmasi și colab., 2022)
$CoFe_2O_4$/g-C_3N_4 (100 mg)	100 mL (Metilorange, 10^{-5} M) – Lumină Vis 300 W Xe	98 %	(Ismael și Wark, 2022)
$LaFeO_3$/g-C_3N_4 (20 mg)	50 mL (Reactiv negru 19, 60 mg/L) – Lumină Vis 1000 W	97 % - 0,0239 min^{-1}	(Hu și colab., 2023)
U-BFO/OCN (30 mg)	50 mL (RhB, 10 mg/L) – Lumină Vis 300 W Xe	88.5 % - 0,096 min^{-2}	Această teză
U-BFO/PCN (30 mg)	50 mL (RhB, 10 mg/L) – Lumină Vis 300 W Xe	99.2 % - 0,253 min^{-1}	Această teză

Capitolul VI

Al șaselea capitol „**Contribuții personale, concluzii și cercetări viitoare**" evidențiază contribuțiile majore ale cercetării, oferă concluzii importante care reflectă obiectivele tezei și încapsulează direcțiile potențiale pentru investigațiile viitoare.

În cadrul acestui studiu, au fost realizate contribuții semnificative prin sinteza de semiconductori g-C_3N_4 mono-dopați cu oxigen (O) și fosfor (P) în proporții diferite și evaluarea performanțelor lor de fotodegradare sub lumină vizibilă. Infuzia de oxigen și fosfor a îmbunătățit considerabil proprietățile catalizatorilor g-C_3N_4. Compozitele heterojoncționale integrate cu α-Bi_2O_3 și α-Fe_2O_3 în O-g-C_3N_4 și P-g-C_3N_4 au fost investigate, demonstrând caracteristici optoelectronice superioare și eficiență de degradare îmbunătățită. De asemenea, a fost studiat impactul configurațiilor morfologice diferite ale $BiFeO_3$ asupra structurii și activității fotocatalitice a compozitelor. Compozitul $BiFeO_3$/P-g-C_3N_4 cu formă eliptică a demonstrat o eficiență remarcabilă de degradare a RhB, urmând un mecanism de transfer al schemei Z cu radicali superoxid și hidroxil ca specii active principale. Compozitele au menținut o fotostabilitate și fezabilitate economică impresionantă după patru cicluri de degradare.

În concluzie, pe lângă contribuția la progresul fabricării materialelor compozite, cercetarea stabilește un fundal ferm pentru dezvoltarea materialelor fotocatalitice avansate capabile să abordeze provocările presante de mediu cu eficiență și precizie sporite. Mergând mai departe, perspectivele obținute din această teză de doctorat deschid calea pentru modele compozite mai avansate și pentru dezvoltarea de materiale ingenioase adaptate pentru aplicații specifice foto-induse, inclusiv remedierea mediului și conversia energiei.

Sistemele heterojoncționale de tip Z-scheme bazate pe O și P-mono-dopat-g-C_3N_4 cu diferiți fotocatalizatori de tip oxid metalic au îmbunătățit semnificativ eficiența de degradare a RhB sub lumină vizibilă, dar există încă un potențial semnificativ pentru investigații suplimentare. Se recomandă sinteza de sisteme ternare de heterojoncțiune g-C3N4 dopate cu P, utilizând catalizatori precum α-Bi_2O_3 și α-Fe_2O_3, α-Bi_2O_3 și $BiFeO_3$, sau α-Fe_2O_3 și $BiFeO_3$. Co-doparea O/P a g-C_3N_4 poate modifica structurile electronice, iar compozitele co-dopate ar trebui evaluate în diverse aplicații fotocatalitice. Cercetările viitoare ar trebui să investigheze performanța acestor compozite sub lumină vizibilă, testându-le în descompunerea apei pentru producția de H_2, reducerea CO_2 și sinteza organică. În plus, compozitele bazate pe P-g-C_3N_4 cu perovskiți precum $CaTiO_3$, $LaFeO_3$ și $BaZrO_3$ și oxizii lor bimetalici trebuie sintetizate și comparate cu performanța compozitelor pe bază de $BiFeO_3$.

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