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Evaluation of synergistic effect of Pd/C doped polyaniline catalyst and its antioxidant efficacy

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An easily recoverable PANI/PdC catalyst was synthesized and thoroughly characterized using XRD, FTIR, and SEM techniques. Its catalytic efficiency was tested in Suzuki coupling, Dehydration, and Buchwald coupling reactions under varied parameters such as time, temperature, and catalyst loading. The catalyst facilitated the high-yield synthesis of biphenyl, benzaldehyde, and N-alkylated products with minimal experimental optimization. PANI/PdC demonstrated a strong synergistic effect, outperforming PANI alone in all three reactions. Moreover, it exhibited excellent antioxidant activity (82% at 3 mg/mL), highlighting its potential as a multifunctional material for industrial and biomedical applications.

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1. Introduction

The role of catalyst in the field of chemistry is held to be pivotal, facilitating in almost every chemical reaction that have commercial and industrial significance. The mechanism of catalyst towards accelerating the reaction via reducing the activation energy and by transforming the action mechanism involved, without altering its chemical integration and not being consumed during the phase of the reaction, thus could be utilized for the forthcoming cycles of reaction. The categorization can be broadly attributed as a single and multi-phase catalyst. With a growing number of homogeneous catalysts and their industrial application have gained a broad array of acceptance with regards to their selectivity and efficiency pertaining to the nature of the catalysts. Also, their portion or quantity involved in these reaction mechanisms appears much smaller compared to its originally expected ranges in the overall output of the chemical reactions [1]. The major disadvantage in the case of homogeneous catalyst is primarily involving the tedious nature for separation of product from the reaction mixture at the end of the reaction mechanism, as the practical catalyst needs to be extracted from the mixture, which requires expensive techniques. In order for devising a suitable cost-effective approach to overcome this complication requires immobilization of soluble catalyst into an insoluble matrix, typically using simpler protocol polymers [2,3]. Multiphase catalysts are relatively cheap compared to single-phase catalysts in dissimilarity, and also promote fast product isolation without boring workup. Multiphase polymer assisted catalysts have been extensively studied in recent years, in particular on conjugated polymers [4-9].

Polyaniline is one of the extensively studied materials which acts as conducting polymers employed for electronic and optical applications and also its environmental sustainability and with effective redox properties, but only there are few literatures available in the utilization of uncharacterized PANI doped metal catalysts [9-11]. The present study conceived this idea in employing interactive PANI as support that immobilizes the metal complex securely without disintegration during the catalytic reactions due to its extremely conductive and redox properties. Additionally, the implication of easy to prepare protocols from the cheap starting material

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(aniline), manageable doping levels via acid doping or base de-doping process, non-solubility, and inert nature in most of the organic solvents with water being quite essential in their application as multi-phase catalyst support [12-14]. Suzuki coupling is achieved in the presence of ligands namely: phosphorous and acetate involving single-phase palladium catalyst. Over the years, there are several attempts taken towards enhancing the catalytic activity with PANI/PdC, however, the limitations are gravely been associated with regards to separation of catalysts, product toxicity, and contamination, thus resulting in researchers to investigate the use of heterogeneous catalysts [13, 14].

The limited supply of water-soluble aryl halides, however, in the absence of additives limits the reach of Suzuki coupling under ligand-free conditions. Succeeding in these boundaries requires the introduction of the Suzuki coupling by imparting newer approaches. Recently, studies on Suzuki coupling have been documented that the application of polyaniline doped palladium at different concentrations, along with the phase transfer catalyst. The catalyst could serve as an alternative for achieving better yields [15-17]. In this research, Suzuki coupling reactions were performed without the use of any phase transfer catalyst and improved yield and efficiency. In present study, reported the PANI/PdC catalyst synthesis and its characterization via XRD, FTIR, and SEM. A reaction mechanism involving Suzuki coupling, Buchwald coupling, and selective oxidation are essential reaction mechanisms in advanced organic synthetic chemistry. These reactions collectively were performed via utilization of presently synthesized and characterized Polyaniline doped with palladium-carbon (PANI/PdC) as an effective catalyst and their catalytic ability in terms of their efficiency was compared with un-doped PANI as a catalyst. The impact of various parameters such as temperature, catalyst loading, and reaction time on the reactions was examined to assess the efficiency of the catalyst in a given reaction and to determine the catalytic effect in a given reaction.

2. Materials and methods

2.1. Materials

All reagents were Analytical grade. Bromobenzene, 1-amino-3-chlorobenzene and benzyl alcohol, N-Methyl piperazine, Aniline, Ethanol, potassium dichromate (K_2Cr_2O7), Palladium/carbon (Pd/C), Potassium carbonate (K_2CO_3), potassium tertiary butoxide, acetonitrile, hydrogen peroxide (30%) solution were acquired from Merck. Phenylboronic acid, sulphuric acid and 1,4-dioxane from sigma Aldrich and Merck purchased. All reagents were weighed and handled in air.

2.2. Synthesis of polyaniline

A mixture of aqueous sulphuric acid (49.7 mmol in 17.7 ml of water) was taken into a beaker and added distilled aniline (4.63 g, 42.9 mmol) drop wise at 25-30°. The reaction mass stirred for 20 minutes. The freshly prepare solution of potassium dichromate (0.9 g) was dissolved in demineralized water (25ml) was added into reaction mass at room temperature and stirred for two hours. At the end of the reaction, a dark green color solution of polyaniline was precipitated, which was filtered and washed with demineralized water (100 ml) followed by methanol (25 ml), the crude synthesized polyaniline mass was dried at room temperature.

2.3. Preparation of PANI/PdC mixture

To synthesize PANI/PdC, 0.9g of polyaniline, 15 mL of ethanol and 1.0g of palladium-carbon were taken in a beaker. The mixture was heated to 80°C, and continuously stirred the reaction mass until the ethanol was completely evaporated at 80°C. The obtained precipitate was filtered, isolated and dried at 55-60°C in an oven to get pure PANI/PdC (1.6 g), under nitrogen atmosphere.

2.4. Characterization techniques

2.4.1. X-ray diffraction analysis

The XRD (X-ray diffraction) explored were performed using a Rigaku X-ray diffract meter powder X-ray meter). The XRD analysis were recorded in the 2θ range from 10° to 80° with step width 0.02 degree and step time 1.25 second using Cupper Kα radiation (Lamda = 0.15418 nm). The XRD peaks were analyzed by the standard pattern provided by JCPDS file. Complies with the observed peaks with (Model: PW1710).

2.4.2. Fourier transforms infrared spectroscopy analysis

Using Perkin Elmer FT-IR spectrometer in the frequency range of 4000 cm^{-1} - 600 cm^{-1} with a resolution of four cm^{-1}, eight scans. The small quantity of the PANI and PANI/PdC catalyst was mixed together with potassium bromide powder and pelletized to register the FTIR spectrum. Blank potassium bromide pellet was used for background edited The FTIR analysis of the PANI and PANI-PdC was recorded.

2.4.3. Scanning electron microscope and Nuclear magnetic resonance spectroscopy

Themorphological study of PANI and doped PANI/PdC catalyst was performed using scanning electron microscopy carlzeiss supra-55, Germany operating at 20 Kv (SEM Model: JEOL JSM 6360). The H^1 NMR spectrum of the product of coupling reactions was recorded using bruker-400MHz NMR system in $CDCl_3$.

2.5. Antioxidant activities

The in vitro antioxidant activities such as DPPH radicals scavenging effect of PANI and doped PANI/PdC was performed by standard protocol as described by Alipour et al. [18].

3. Results and discussion

3.1. XRD studies of PANI and PANI/PdC

In his analysis, the standard XRD analysis of PANI and PANI/PdC catalyst has shown in Fig. 1 and 2. The spectrum XRD analysis of PANI (polyaniline) in Fig. 1 observed two softly peaks, determined at 20° and 25° the circle planes of benzol rings in near chains or the proximity contact inter-chain distance by the XRD peak at 20° indicates the characterization [19]. The peak indicated around 25° possibly decided to the spreading from PANI chains at inter-planar spacing and the very poor concentration of the observed peak confirmed that the PANI have an amorphous nature with less crystalline [20,21]. The XRD analysis of synthesized PANI/PdC catalyst is revealed in Fig. 2. X-ray diffraction analysis peaks were observed at 24.19°, 29.92° and 40°. The XRD analysis of wide peak fixed at 25° possess a hump at 20° is because of the crystalline PANI of area [22, 23].

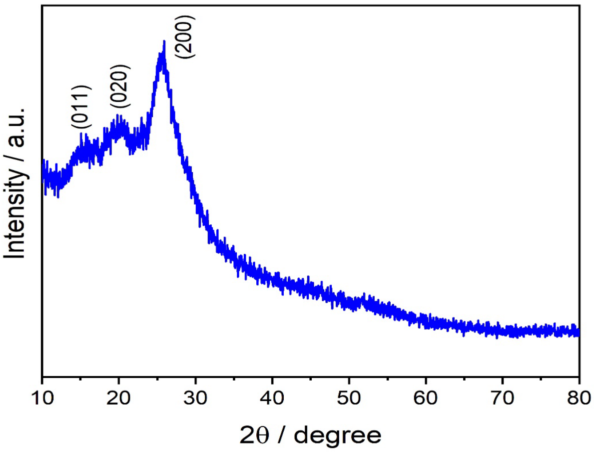

Fig. 1. X-ray diffraction pattern of pristine PANI.

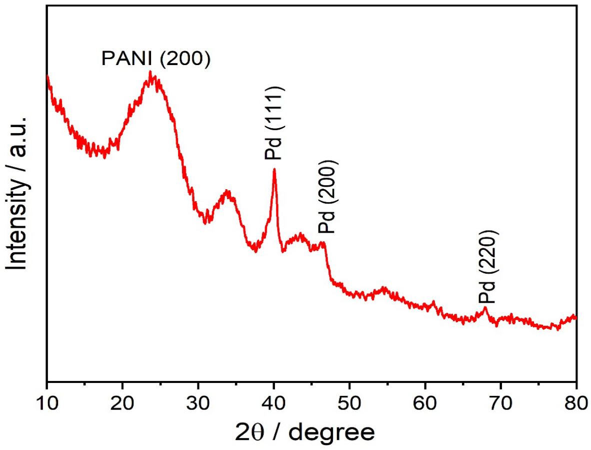

Fig. 2. XRD pattern of PANI/PdC.

3.2. FTIR studies of PANI and PANI/PdC

The FTIR spectrum of PANI, PANI/PdC catalysts was display in Fig. 3 and Fig. 4. The FT-Infrared analysis of PANI shows the broad vibrations at 2355-2752 cm^{-1} is owing to phenyl Carbon-Hydrogen stretching vibrations similar to the vibration band at 2935-3301 cm^{-1} is because of Nitrogen-Hydrogen stretching of phenyl amines. The low-wavelength vibration at 1548 cm^{-1} has a main circulation from the quinoid cyclic, while the higher wavelength mode at 1483 cm^{-1} displays the existence of benzenoid cyclic unit. The exist of bands in the order of 1451-1601 cm^{-1} are ascribed to asymmetric Carbon-six ring stretching modes (Fig. 3 and 4). The Carbon-Nitrogen stretching vibration was displayed a band at 1317 cm^{-1}. In plane bending vibration in Carbon-Hydrogen occurs at 1086 cm^{-1}. The absorbance bands at 510 & 819 cm^{-1} are owing to the vibration of bending of Carbon-Hydrogen out of plane and p-bi substituted aromatic rings, individually [24-26]. Characterization of vibrations in the area of 1000-1501 cm^{-1}. PANI displayed 510, 819, 1150, 1310, 1475 and 1548 cm^{-1} are characterization of vibrations is bands. FT-Infrared analysis of the synthesized PANI/PdC catalyst is displayed in Fig. 4. The best essential quality vibration bands of PdC/PANI catalyst was noticed at 3453 cm^{-1} (Nitrogen-Hydrogen stretching), 1620 cm^{-1} (vibration of quinoid stretching), 1490 cm^{-1} (vibration of Benzenoid stretching), 1241 cm^{-1} (2nd C-N stretching vibration) [27], 1023 cm^{-1} (anion dopant of vibration band) and 788 cm^{-1} (p-bisubstituted aromatic rings) 1890 cm^{-1} also the changing of Nitrogen=Q=Nitrogen stretching (unit of quinoid) and Nitrogen-B-Nitrogen stretching vibration (unit of benzenoid) towards decreased frequencies are because of the interaction between PANI and Pd [23].

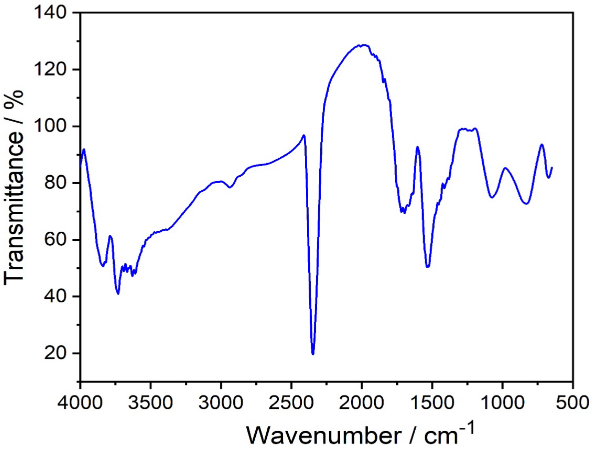

Fig. 3. FTIR spectrum of the pristine PANI.

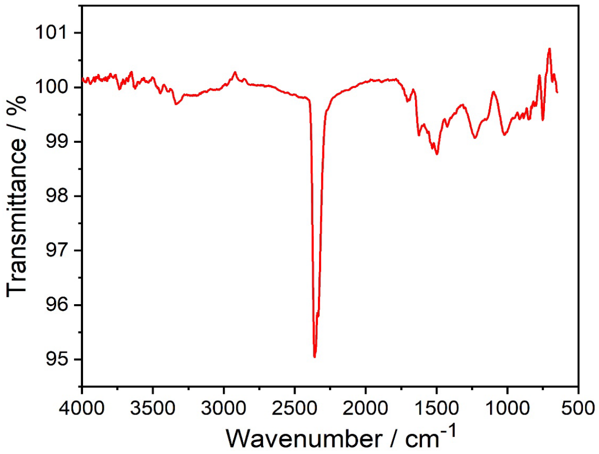

Fig. 4. FTIR spectrum of the PANI/PdC.

3.3. Surface morphology of SEM micrographs studies

Part a and b of Fig. 5, the PANI and PANI/PdC catalysts display the SEM images and the pure PANI displays 0.001 mm-sized irregular stratified morphology as depicted in Fig. 5. The PdC particles are often depicted on PANI sheets as displayed in Fig. 6. It is determined that the circulation of PdC particles on the PANI matrix is consistent and the sizes of PdC particles square measure measured as 1, 2 and 100 µm as indicated by the parallelogram (Fig. 5 and 6).

3.4. Effect of various parameters on PANI/Pd/C catalyzed selective dehydration

The phenyl methanol dehydration reaction was carried out using H_2O_2. It is used as an oxidizer in the presence of 0.005 g of PANI/PdC in acetonitrile. Instead of acetonitrile, dehydration of phenyl methanol was also studied using different solvents H_2O, 1,4-dioxane (45 %), THF (25 %), and toluene (less than 5 %) [13-15]. Based on the yields, acetonitrile was found to be the best solvent, while toluene and THF yielded very poorly. Dehydration of phenyl methanol is also achieved by replacing PANI/PdC with PANI resulting in low yield [16, 17]. The impact of different parameters such as load of catalyst, reaction mass temperature, and time on dehydration of phenyl methanol is shown in Table 1. The reaction scheme is given below.

Selective phenyl methanol dehydration is carried out with a different percentage (% w/w) of the loading catalyst, such as PANI/PdC, relative to PANI. The percentages of the loading catalyst and the yields obtained are shown in Table 1. Dehydration of phenyl methanol in the presence of PANI/PdC (Entity:1,2,3,4,5) yield is the better overall percentage of catalyst loading except at 0.002 g; while dehydration of phenyl methanolin the presence of PANI catalyst yields lower even in higher catalyst loading at 0.025 g. The explored catalyst output equivalent from 0.025 to 0.005 g relative to starting material (1 mmol) resulted in the high conversion of phenyl methanol to benzaldehyde and higher yield (0.005 g). The catalyst quantity of more than 0.005 g decreased benzaldehyde yield in mild conditions due to lower activation energy as catalyst concentration increased to benzaldehyde and benzoic acid formed due to benzaldehyde yield was decreased [16]. On the other hand, when less than the catalyst quantity of 0.005 g utilized as a catalyst concentration decreased due to increased activation energy to give benzaldehyde and unreacted benzyl alcohol. More than or less than 0.005 g of catalyst, the yield was affected (Table 1).

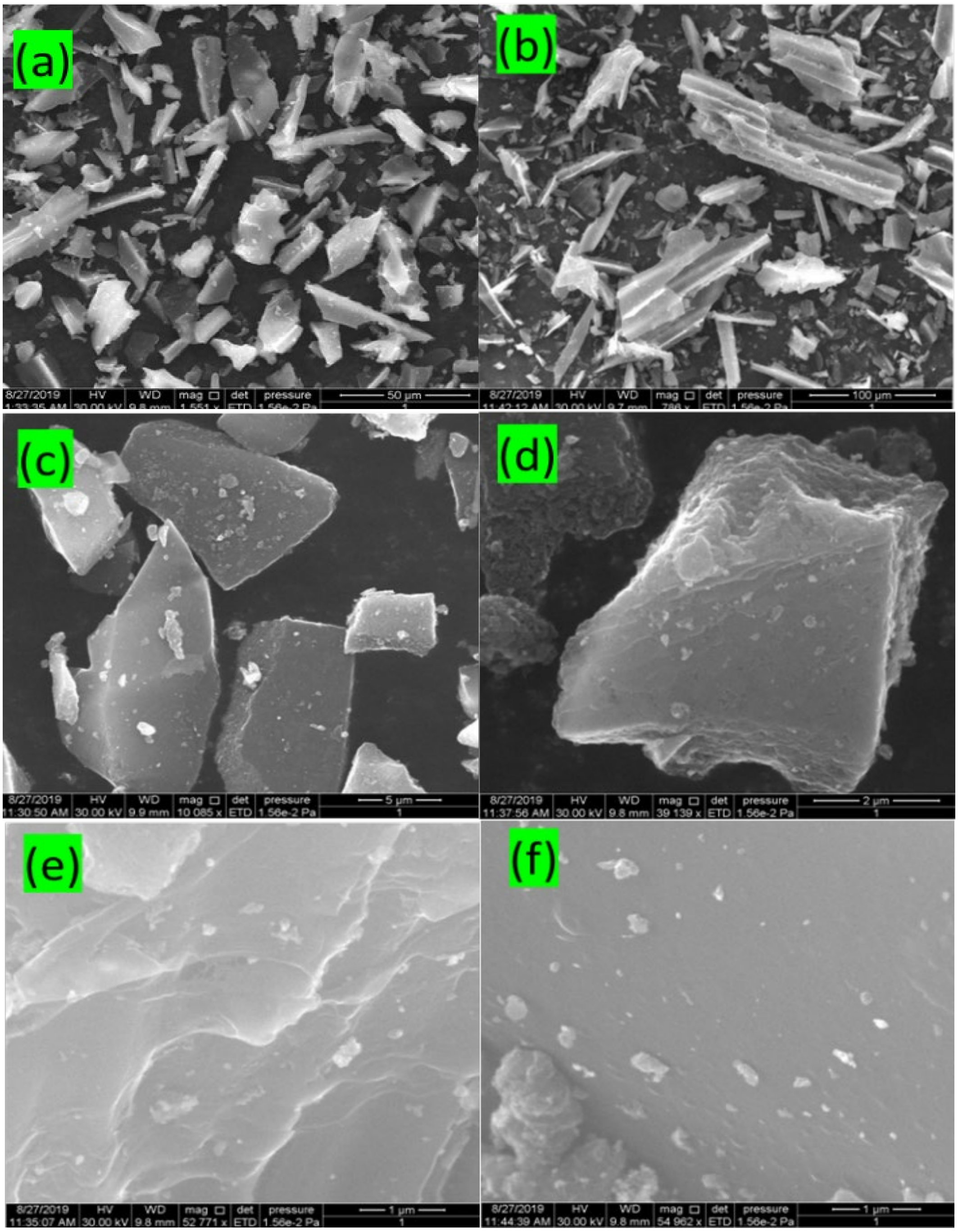

Fig. 5. (a-f) SEM image with different magnifications of polyalanine.

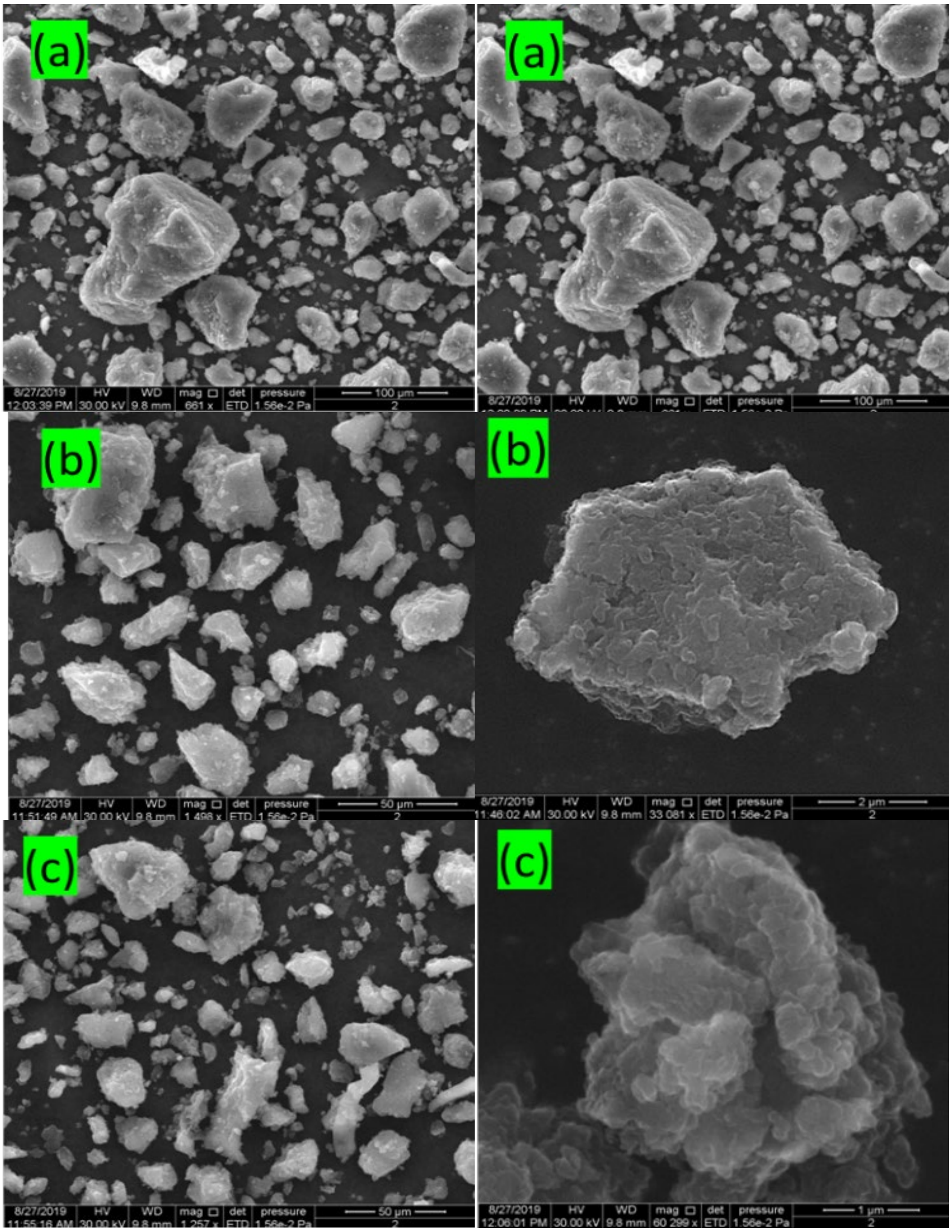

Fig. 6. (a-f) SEM images with different magnifications of PANI/PdC.

The effect of reaction time with regards to the yield involving acetonitrile solvent with PANI/ PdC catalyst showcased that the best yield at 24 hours was determined. Steep declination in the yield was observed when the reaction was performed under lesser time interval (i.e.) <24 hours. Benzyl alcohol dehydration performed with PANI in place of PANI/PdC resulted in very low yields even after maintaining for 24 hrs [8-10]. Table 1 provides the effect of time and related yields achieved. The reaction time output from 4 hrs (20 %) to 24 hrs (92 %) resulted in longer hours of conversion more benzyl alcohol into benzaldehyde was determined. The reaction time decreased as well as the conversion of aldehyde decreased [11, 12]. The reaction mass was maintained for 4 hours (20%) in a mild state created lesser yield due to the lower solubility of the catalyst. On the other hand, the reaction time substantially increased 24 hrs achieved more yield (92%) due to more solubility catalyst [11, 18, 20].

Similar to the effect of temperature observed, based on the research investigated the effect of reaction temperature on yield via employing acetonitrile solvent, for which the best yield was obtained at 80°C in presence of PANI/PdC. The reactions carried out below 80°C, the lesser yield was observed. The dehydration of phenyl methanol which is observed from the catalytic activity of PANI with results showcasing a lesser yield even at higher temperatures of 80°C. The effect of temperature and corresponding yields are shown in Table 1. Phenyl methanol dehydration has been analyzed at various temperatures from 25 to 80°C (Fig. 6 and 7). The yields steadily increased upon careful analysis of the data with the reaction mass temperature was gradually raised from 25°C to 80°C. The maximum conversion (benzaldehyde) rate of 91% was attained at a reaction mass temperature of 80°C. The key reason for improved benzaldehyde conversion and yields at a high temperature as a result of increased agitation followed by increased molecular movement facilitates the OH radical species and phenyl methanol to accumulate. The reduced in benzaldehyde selectivity above 80°C, due to faster degradation of H_2O_2 in aqueous medium under higher temperature with decreased oxidizing effect. The reaction was conducted less than or more than that of 80°C yield was affected (Fig. 6 and 7).

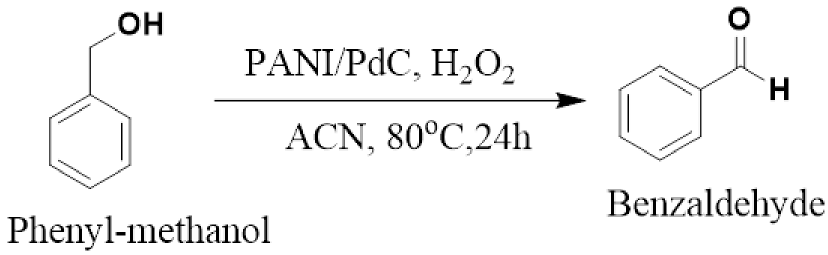

Fig. 7. Dehydration of benzyl alcohol with hydrogen peroxide.

Its catalytic efficiency was explored after a thorough characterization of the synthesized catalyst. Chemical effects emerge from the formation of radical organisms, due to homiletic molecular breakage [24]. Hydroxyl-radical species (HO•) are produced in the case solvent of water. These (HO•) radical are heavy oxidants and are fastly reactive to organic compounds [28]. In the pharmaceutical industry, fragrances, dyes, and agricultural industries, benzaldehyde is an important organic intermediate. 14 principles chemistry was designed in search of more sustainable chemistry [29] and recommended the use of securely auxiliaries and organic solvents in synthesis. The application of toxic and contaminate oxidizers [30] in organic chemistry should be changed by harmless, environmental healthy, and abundant oxidizer. A few environment-friendly controlled studies have therefore concentrated this synthesis of benzaldehyde utilize H_2O_2 as a green oxidizer [31-34]. Sonochemistry has confirmed a hopeful technique for reducing the number of chemicals, minimizing the length of the reaction, avoiding extreme conditions, and contributing to energy savings [35]. In this analysis, expected to easy conditions for converting phenylmethanol to benzaldehyde in the presence of PANI/PdC using H_2O_2 (30 %) in acetonitrile. Catalytic activity for PANI/PdC has been investigated. In the beginning, the accuracy of the reaction conditions appeared to be important for phenyl methanol dehydrogenation. Consequently, the effect of catalyst load, solvent, temperature, and reaction time on a model reaction dehydrogenation of benzyl alcohol with hydrogen peroxide varied in various reaction conditions. And without catalyst and H_2O_2 (30 %), the conversin was found to fail [21-25].

Best solvent result was obtained with acetonitrile. After checking various oxidizing agents such as H_2O_2 (82%), tertbutylperoxide (40%), perchloric acid (less than 10%), the best oxidizing agent was found to be H_2O_2. Finally, after various percentage loadings, 0.005 g of PANI/PdC was found to be the optimum for 24 hours at 80°C in the complex and the highest yield obtained (92%). TiO_2-supported catalysts comprising Pd–Zn are not normally connected with dehydration reactions. The present employment illustrates this by cautiously develop the catalyst for PANI/PdC. Similar catalytic quality results can be obtained with one percent already Au/Pd/TiO_2 catalysts studied. The preparation of 1 % Au-Pd-TiO_2 catalyst, using the adapted impregnation technique and analyzed under the same conditions gave Benzaldehyde a 56% conversion with 74% selectivity. This PANI/PdC catalyst showed activity with 93 percent benzaldehyde selectivity. This shows that on Pd-based monometallic designed catalysts for the dehydration from phenyl methanol to benzaldehyde, PANI may be a perfect and low-cost substitute for Au [35].

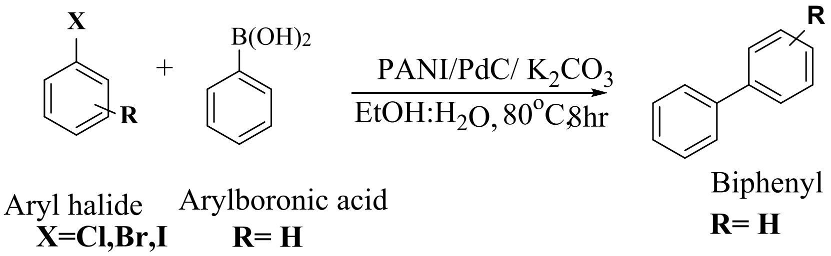

Fig. 8. Suzuki coupling of aryl halide with phenylboronic acid.

Many organisms depend on oxidation to produce the energy needed to power their metabolic functions. However, the start of numerous illnesses, including, rheumatoid arthritis, cancer and atherosclerosis, as well as the deterionation processes related to ageing, are influenced by the unchecked generation of oxygen-derived free radicals [36]. Synthetic antioxidizers are presently employed in industrial processing to minimize harm to human health. The most widely utilised antioxidants, however, have been linked to carcinogenesis and liver damage [37]. In the present study, the antioxidant activity was noticed dose dependent (Fig. 9). From the results of PANI/PdC, the superior activity was found in 3mg/ml followed 2.5 and 2 mg/ml for 81, 68 and 48% and the minimal activity was noticed in 1mg/ml for 12% respectively. Additionally, the BHT has showed better DPPH radical scavenging activity than sample for 94% in 3 mg/ml respectively (Fig. 9). The present study results matched with previous study of Gizdavic-Nikolaidis [38], reported the better DPPH radical scavenging effect of aniline and polyaniline. The development of the dimeric species should be the stage that moves along the slowest, according to Gospodinova and Terlemezyan [39], the trimers are produced when the dimers undergo quick oxidation, deprotonation, and electrophilic aromatic substitution with an aniline monomer after being formed.

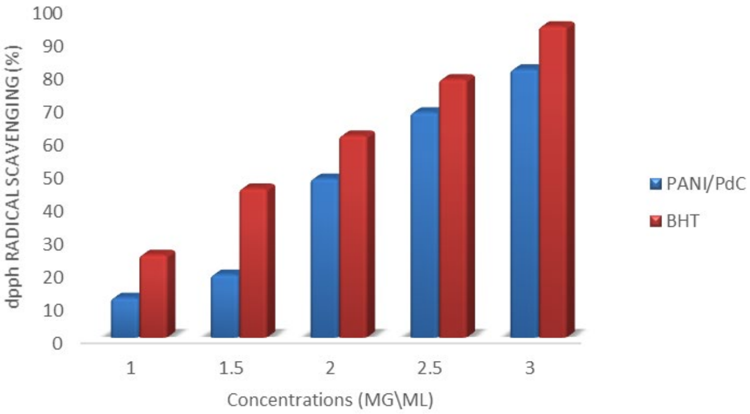

Fig. 9. DPPH radical scavenging effect of PANI/PdC.

4. Conclusion

The above chemical synthesis mechanism which involves the assisted PANI/PdC complex showed a synergistic effect in catalysis of benzyl alcohol dehydrogenation with H_2O_2, coupling reactions of bromobenzene with phenylboronic acid and tert-butyl (3-chlorophenyl) carbamate with N-methyl piperazine. PANI/PdC catalyst was successfully synthesized and characterized and the use of the synthesized supported PANI/PdC complex as a heterogeneous oxidation catalyst for performing Suzuki coupling, and Buchwald Hartwig coupling reaction can simultaneously provide high final product yield, selectivity, easy catalyst separation and adequate recycling reaction efficiency. The catalytic efficiency of PANI/PdC was observed to have a significant impact with superior yield from the observed reaction mechanism which was further then compared under various time intervals and temperature ranges and also it's a best radical scavenger for food applications.

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