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ADSORPTION CAPACITY, KINETICS, AND APPLICATION OF MOLECULARLY IMPRINTED POLYMERS DI-(2-ETHYLHEXYL) PHTHALATE AS ADSORBENTS IN SOLID-PHASE EXTRACTION METHOD

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ABSTRACT

The purpose of this research is to synthesize, characterize, optimize, and apply MIP as an adsorbent material for solid phase extraction (SPE). The synthesis method used is precipitation polymerization. MAA as a monomer was combined with EGDMA as a crosslinker and used DEHP as imprinting molecules. Structural characterization of MIP using SEM, and TGA. MIP optimization using time and concentration parameters was carried out to test MIP's ability to adsorb DEHP. The MIP_DEHP_MAA-co-EGDMA or MIP_DEHP obtained were white powders. The MIP surface morphology is granules with shape, and size more uniform than NIP. The MIP_DEHP adsorption ability on the time effect reached optimum at 90 minutes. The adsorption kinetics model for MIP_DEHP follows the kinetic model of pseudo-second-order (PSO) adsorption. The adsorption ability to the effect of concentration increases with increasing concentration. The adsorption capacity value of MIP_DEHP was 4.3917 mg/g determined according to the Langmuir isothermal adsorption model. The application of MIP_DEHP as an adsorbent in the SPE method has proven to be very good and allows it to be reused because the reusability percentage is very high, namely 95.52%.

Keywords: Synthesis, Polymers, MIP, DEHP, Adsorption.

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INTRODUCTION

Phthalates are a chemical group used as plasticizers in many industrial products such as medical equipment, children's toys, and food and beverage packaging.[1,2,3] Polyethylene terephthalate (PET) plasticizers group are commonly used in the packaging industry.[4] They can contaminate food or beverages[2,3,4], especially if stored for a long time, used repeatedly, and in contact with acidity and high temperatures.[5] The type of PET that is often used is di-(2-Ethylhexyl) phthalate (DEHP)[6,7] because DEHP is known to have strong and water-resistant characteristics, but these compounds can be easily released when stored for a long time in hot conditions[7] and the toxicity properties of phthalates cause health problems[3,8] so that needs to be detected.[9] DEHP can be performed by High-Performance Liquid Chromatography (HPLC)[10], and SPE coupled with LC-MS/MS.[11] The advantages of SPE are the use of less solvent, efficient time, few steps, and low cost.[12] The polymer used as a highly selective adsorbent for the target in SPE, namely Molecularly Imprinted Polymers (MIP).[13] MIP has resistance and high stability to temperature, pressure, and pH[14] and can be reused.[15] Phthalate compounds can be used as templates and target compounds in MIP.[16] MIP DEHP are made by reacting monomer, crosslinker, porogen solvent, and DEHP as a template molecule. The DEHP MIP cavity formation follows the molecular structure, size, binding site, and functional groups of DEHP.[17] The monomer commonly used in the synthesis of MIP is methacrylic acid (MAA). Its ability to interact with molecules and its adsorption is relatively better than other types of monomers.[18,19] The MAA in MIP is applied as a cholesterol sensor[20] and it is also possible to use it as a DEHP sensor. The combination of ethylene glycol methacrylic acid (EGDMA) and MAA resulted in a stable and relatively rigid MIP.[20,21] The polymerization method commonly used in MIP synthesis is bulk polymerization, but agglomerates are formed[22] and easily damaged.[23] Another polymerization method is precipitation with better analytical

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performance,[24] no agglomeration, uniform spherical particles, more number, and minimal solvent.[25] The adsorption process of MIP could be well explained through the Langmuir isotherm model and the PSO kinetic model.[26] This study aims to synthesize, characterize, and optimize dMIP as a DEHP adsorbent using a combination of MAA and EGDMA using precipitation polymerization. The morphology of the synthesized MIP was determined by Scanning Electron Microscope (SEM), changes in the decomposed mass by a Thermogravimetric Analyzer (TGA), and then applied to the SPE method. Before being applied in the SPE method, MIP determined its kinetics and adsorption capacity for DEHP compounds.

EXPERIMENTAL

Materials and Methods

The chemicals used include DEHP 99.5% (Sigma-Aldrich), EGDMA (Sigma-Aldrich), methacrylic acid (MAA) 99% (Sigma-Aldrich) as a monomer, 2,2'-azobisisobutyronitrile (AIBN) (Sigma Aldrich), acetone pro analyst (Merck), toluene pro analyst (Merck), methanol Grade HPLC (Merck), acetic acid pro analyst (Merck), nitrogen, aqua dest, Whatman paper no. 41. The equipment used included glassware, sonicator, analytical balance, vortex, centrifuge, shaker, oven, water bath, vial, 100 L and 1000 L micropipette, spectrophotometer UV-Vis (Shimadzu, UV-2600), Thermogravimetric analyzer (TGA) NEXTA STA (Hitachi STA200RV with Real View Sample Observation), SEM (JEOL, JSM-6510 LA).

Experimental

Several stages in this study consist of MIP and NIP synthesis, MIP characterization, MIP optimization (time and concentration effect), capacity determination of capacity, and adsorption kinetics.

Synthesis of MIP and NIP

The pre-polymerization stage was carried out by reacting MAA 4 mmol and DEHP 1 mmol for 5 minutes, then adding EGDMA 8 mmol and toluene was added and allowed for 5 minutes. Then sonicated and degassed using N_2. Then it was reacted with AIBN 1 mmol, sonicated, and degassed with N_2. The polymerization step was carried out at 60ºC for one day.[27] The polymerization results were washed using acetone, methanol, and aqua dest and obtained MIP, namely MIP_DEHP$_{(Before\ Extraction,\ BE)}$ or MIP_DEHP$_{(BE)}$. Then, the DEHP compound was extracted for 30 minutes from MIP utilizing an eluent composition of acetic acid: methanol (1: 9 v/v) by sonication technique, and the MIP obtained was named MIP_DEHP$_{(After\ Extraction,\ AE)}$.[28] The extracted DEHP was identified using a spectrophotometer UV-Vis, and the obtained MIP_DEHP$_{(AE)}$ was rinsed with methanol and aqua dest until neutral pH, dried, and characterized. In contrast, a nonimprinted polymer (NIP) or NIP_MAA-co-EGDMA was synthesized without utilizing DEHP and extraction processes.

Time Effect on Adsorption Ability of MIP

Amounts of 5 mL of 10 mg/L DEHP standard and 50 mg of MIP were put into seven vials. Next, the mixture was shaken based on time variations of 10, 30, 60, 90, 120, 150, and 180 minutes. After the adsorption process, the filtrate containing DEHP was determined using UV-vis. The study of the adsorption kinetic was described using kinetic models of pseudo-first-order (PFO) and PSO.[29]

Concentration Effect on Adsorption Ability of MIP

Amount of 5 mL of DEHP with concentrations variation of 1, 5, 10, 15, 20, and 25 mg/L at pH 6 to 7 mixed with 50 mg of MIP contained in vials. The mixture was shaken and filtered at room temperature and optimum time, then analyzed with UV-Vis at maximum wavelength.[28]

Determination of MIP Adsorption Kinetics

The adsorption kinetics can be determined from the time effect analysis data using equations of PFO and PSO.

MIP Adsorption Capacity Determination

The adsorption capacity can be determined from the concentration-effect analysis data using the Freundlich adsorption isothermal equation and the Langmuir isothermal.[30] The Freundlich and Langmuir isotherms parameters can also be used to analyze the adsorption mechanism.[31]

Application of MIP as an Adsorbent (Stationary Phase) using the SPE Method

MIP-SPE Cartridge Preparation

A total of 30 mg of MIP is loaded into the SPE cartridge equipped with a filter. Then, eluted with methanol solvent to activate the cartridge. The cartridge that has been connected to the SPE vacuum device is set at a specific pressure so that the solution drop rate is one drop per 2 seconds.

SPE Process

The amount of 5 mL of DEHP solution with six different concentrations of 9, 15, 18, 21, 24, and 27 mg/L was put into the cartridge, and then the filtered solution was analyzed utilizing UV-vis at a maximum wavelength of 256.2 nm. Furthermore, 5 mL of methanol was passed through the SPE cartridge and the filtrate was analyzed with UV-Vis to determine % reusability.

RESULTS AND DISCUSSION

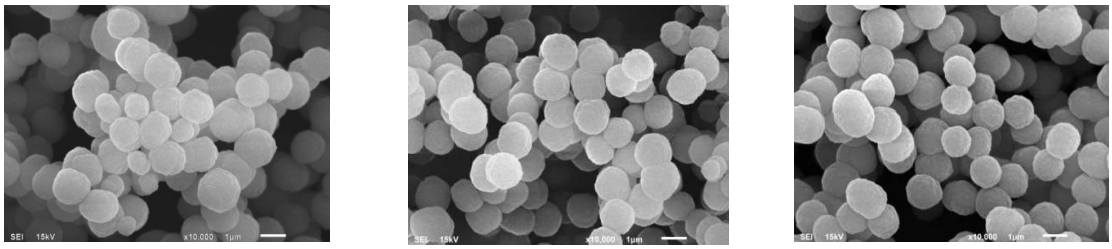
Synthesis of MIP

A polymer in the form of white solids has been produced in this study through a synthesis process using the precipitation method. MAA and DEHP first interact covalently via hydrogen bonding in the toluene solvent in the pre-polymerization step. Furthermore, the initiator assists in the formation of cross-links between MAA and EGDMA in the polymerization step which includes initiation, propagation, or chain elongation and termination steps. The polymer produced from the polymerization process was washed and extracted several times to release the DEHP imprinted molecules in the polymer using sonication to obtain a DEHP molecule imprinted polymer (MIP_DEHP).

MIP Characterization

Characterization of MIP_DEHP by SEM

The morphology surface characterization of NIP, MIP_DEHP$_{(BE)}$, and MIP_DEHP$_{(AE)}$ by SEM are exhibited in Fig.-1.



(a) (b) (c)

Fig.-1: Surface Morphology with 10,000x Magnification (a) NIP, (b) MIP_DEHP$_{(BE)}$, (c) MIP_DEHP$_{(AE)}$

The three polymers have different morphologies including small and non-uniform grain size, less dense density, and coarse texture for NIP. MIP_DEHP(BE) and MIP_DEHP(AE) surface morphology consists of spherical granular, uniform in size, less dense, and a smoother texture than NIP.

MIP Characterization Using TGA-DTA

Thermo gravimetric analysis (TGA) was used to know the stability of thermal and the difference in the decomposition rate of NIP and MIP. Fig.-2 exhibited the thermograms of NIP, MIP_DEHP$_{(BE)}$, and MIP_DEHP$_{(AE)}$. TGA thermograms of NIP and MIP_DEHP$_{(AE)}$ showed a similar pattern because the DEHP compound in MIP had separated from the polymer, while MIP_DEHP$_{(BE)}$ still contained DEHP compounds so the thermogram pattern was different. NIP, MIP_DEHP$_{(BE)}$, and MIP_DEHP$_{(AE)}$ experienced multiple mass losses. The lost mass of NIP begins to occur at a temperature of about 28ºC to 225ºC, MIP_DEHP$_{(BE)}$ starts at around 28ºC to 235ºC, while MIP_DEHP$_{(AE)}$ starts from an ambient temperature of 27ºC to 240ºC. At the initial temperature, it is assumed that the mass loss of water.

The TGA Thermogram of MIP_DEHP$_{(BE)}$ can be seen that there is a peak at a temperature of around 125ºC to a temperature of around 315ºC, and this peak is not found in NIP and MIP_DEHP$_{(AE)}$. The presence of DEHP in MIP_DEHP$_{(BE)}$ is indicated by a peak. The mass loss of MIP_DEHP$_{(BE)}$ starts at about 28ºC, but a huge mass loss occurs at temperatures around 335ºC to 470ºC. NIP_MAA-co-EGDMA, at a temperature

of about 28°C, began to lose mass, but the largest mass loss was at a temperature of around 390°C to 460°C, while for MIP_DEHP$_{(AE)}$, it began to lose mass at around 27°C and the largest mass loss was at a temperature of around 340°C to 460°C. The higher temperature causes the bonds between the monomers to break. Based on the high temperature at the end of the degradation that occurs, MIP is a polymer that is stable to temperature.

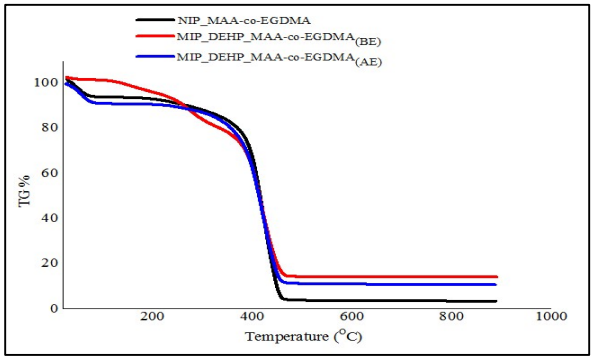


Fig.-2: Thermogram of TGA Results for (a) NIP, (b) MIP_DEHP$_{(BE)}$, and (c) MIP_DEHP$_{(AE)}$

Optimization of MIP Adsorption Ability

Although the adsorption ability of MIP_DEHP$_{(AE)}$ is better than NIP, optimization of MIP adsorption ability parameters such as concentration and contact time must be carried out. Optimization was conducted to determine the kinetics and adsorption capacity of each MIP.

The Time Effect on the Adsorption Ability of MIP_DEHP$_{(AE)}$

The time effect on the adsorption ability of MIP_DEHP$_{(AE)}$ against DEHP can be seen in Fig.-3.

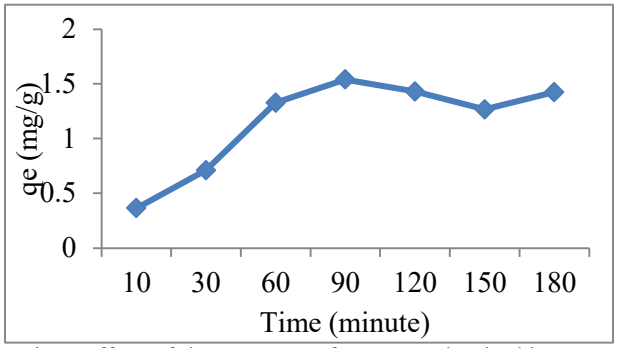


Fig.-3: The Time Effect of the Amount of DEHP Adsorbed by MIP_DEHP$_{(AE)}$

Fig.-3 exhibited an increase in the amount of the adsorbed DEHP compound with increasing contact time until it reaches the maximum adsorption. When maximum adsorption is achieved, MIP is estimated to have been saturated by DEHP, and adsorption ability will tend to decrease the next time. MIP material saturated with DEHP will experience DEHP desorption and then re-adsorb DEHP at 180 minutes so that there is an increase in the value of q_e. Ninety minutes is the best time for MIP_DEHP$_{(AE)}$ to adsorb DEHP in the amount of 1.542 mg/g. The adsorption kinetics model was used to determine the adsorption kinetics of mass transfer and chemical reaction dynamics. The effect of adsorption time data on DEHP by MIP was analyzed by equations of PFO and PSO.

The Concentration Effect on Adsorption of MIP_DEHP$_{(AE)}$

The higher the initial concentration of the DEHP standard solution, the more DEHP can be adsorbed by MIP_DEHP$_{(AE)}$. If equilibrium adsorption has reached the maximum limit, then the adsorption ability of MIP_DEHP$_{(AE)}$ tends to be constant. The correlation between concentration variations and the amount of DEHP adsorbed by MIP can be seen in Fig.-4 that the DEHP amount adsorbed by MIP increased with increasing DEHP concentration in the solution. This indicates that the adsorption process by MIP has not

reached equilibrium. Therefore, the MIP_DEHP$_{(AE)}$ adsorption capacity can be calculated using the Freundlich and Langmuir isothermal equation.

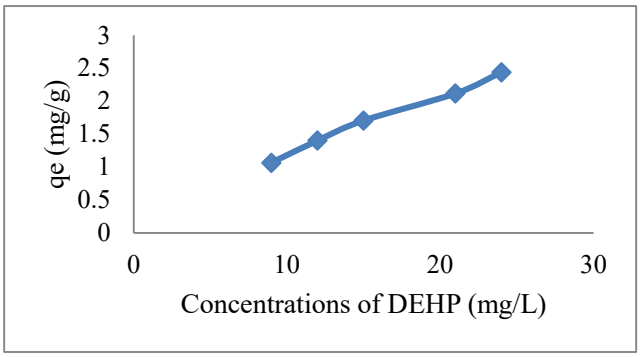


Fig.-4: Concentration Effect on the Adsorption Ability of MIP_DEHP$_{(AE)}$ Against DEHP

Determination of Adsorption Kinetics of MIP_DEHP$_{(AE)}$

Time is an important variable in determining the quality of MIP as an adsorbent. Therefore, the data analysis to determine the adsorption kinetics, which includes chemical reaction dynamics and mass transfer was used in the adsorption kinetics model. Data on the effect of DEHP adsorption time on MIP were calculated using equations of PFO and PSO. The linear regression correlation coefficient values (R^2), PFO kinetic constant (K_1), and PSO kinetic constant (K_2), the qe results of the experimental and calculated are exhibited in Table-1.

Table-1: Parameter Data of DEHP Adsorption Kinetics by MIP_DEHP$_{(AE)}$ using PFO and PSO Kinetic Equations

Type of MIP	q_e (mg/g)			Velocity Constant		R^2	
	PFO count	PSO count	Experiment	K_1	K_2	PFO	PSO
MIP_DEHP_MAA-co-EGDMA $_{(AE)}$	1.206	1.545	1.542	-0.012	0.038	0.641	0.953

Table-1 describes that the DEHP adsorption by MIP_DEHP$_{(AE)}$ for the PSO adsorption kinetics model has an R^2 value closer to 1, which is 0.953 compared to R^2 for the PFO adsorption kinetics model. The qe calculated value based on the kinetics model of PSO for MIP_DEHP$_{(AE)}$ is 1.545 mg/g, closer to the experimental q_e value, which is 1.542 mg/g when compared to the kinetics model of PFO and thus, the synthesized MIP adsorption model follows a PSO adsorption kinetics model with the adsorption rate constant (K_2) MIP_DEHP$_{(AE)}$ was 0.038.

Determination of Adsorption Capacity of MIP_DEHP$_{(AE)}$

The adsorption capacity of MIP_DEHP$_{(AE)}$ was calculated using the Freundlich and Langmuir isothermal model. The appropriate adsorption isothermal model was determined from the linearity of the curve. The linearity of the Langmuir isothermal curve is obtained from the relationship 1/qe and 1/C_e in the Langmuir isothermal equation. In contrast, the linearity of the Freundlich isothermal curve is obtained from the relationship between log qe and log C_e in the Freundlich isothermal equation. The value of the correlation coefficient obtained from the adsorption isothermal Langmuir equation for MIP_DEHP$_{(AE)}$ is 0.989, and the correlation coefficient value obtained from the adsorption isothermal Freundlich equation is 0.976. The R^2 value of the Langmuir isothermal model of MIP_DEHP$_{(AE)}$ was closer to 1 than the Freundlich isothermal model so the adsorption that occurs follows the isothermal model of Langmuir. Therefore, the Langmuir adsorption isothermal model was used to determine the adsorption capacity of MIP_DEHP$_{(AE)}$. In general, the adsorption parameters of each isothermal model are presented in Table-2.

Table-2: DEHP Adsorption Parameters by MIP_DEHP$_{(AE)}$ Obtained from Langmuir and Freundlich Adsorption Isothermal

Type of MIP	Adsorption isothermal of Langmuir			Adsorption isothermal Freundlich		
	b	Q_o	R^2	K_f	n	R^2

MIP_DEHP$_{(AE)}$	0.127	4.392	0.989	2.164	0.605	0.976

Table-2 exhibits that Langmuir's constant value (b) describes the adsorption affinity by MIP_DEHP$_{(AE)}$ or the equilibrium constant is 0.127 L/mg. The value of Q_o MIP_DEHP$_{(AE)}$ is 4.392 mg/g indicating the maximum adsorption capacity of the monolayer where the adsorbent surface is completely covered by a layer of adsorbent molecules and it is concluded that the adsorption capacity value of MIP_DEHP$_{(AE)}$ using Langmuir isothermal is 4.392 mg/g. The surface homogeneity of MIP_DEHP$_{(AE)}$ was shown by experimental data according to the Langmuir model.

Application of MIP_DEHP on the SPE

The MIP_DEHP as an adsorbent was applied on SPE to separate DEHP from the sample solution. The steps used in the SPE process include retention, washing, and elution. In the retention stage, the sample solution was passed on a MIP in the SPE cartridge. DEHP compounds in the solution will be selectively adsorbed by MIP. The DEHP concentration data obtained at the retention stage are shown in Table-3.

Table-3: DEHP Concentration Data Obtained From Several Stages in the SPE Method by MIP_DEHP

No	MIP type	Initial (The initial concentration of DEHP,mg/L)	Retention	Washing	Elution
			$C_{(R)}$ (Retained DEHP concentration, mg/L)	$C_{(R)}$ (DEHP concentration after washing, mg/L)	$C_{(E)}$ (DEHP concentration eluted, mg/L)
1	MIP_ DEHP	15	2.173	12.173	2.057
		21	2.597	18.403	2.442
		27	3.981	23.019	3.788

The DEHP concentration adsorbed by MIP also depends on the MIP concentration used in the separation process using the SPE tool. In this study, the amount of adsorbent (MIP) used was only 30 mg, so not all DEHP compounds could be adsorbed. Next, the washing stage is the stage where compounds that are not adsorbed by MIP will be removed using a washing solvent, namely methanol, and water, and the next stage is the elution stage, which is the stage where the compounds adsorbed by MIP will be removed with the elution solvent. At this stage, 100% methanol was used as the elution solvent because its polarity was lower than the washing solvent used so that the non-polar DEHP that was retained in the MIP could be eluted. Based on data from the stages of the separation process carried out in the SPE method to apply the synthesized MIP as an adsorbent, MIP_DEHP proved to be very good for use as an adsorbent. It is supported by the data from the characterization results and the data obtained from the adsorption ability test of MIP_DEHP against DEHP, indicating that MIP_DEHP has a high capacity and selectivity to DEHP. MIP that has adsorbed DEHP can be reused by conducting a desorption process first to release DEHP contained in MIP. In order to prove that MIP can be reused, it is necessary to calculate the percent recovery from the adsorption-desorption process.

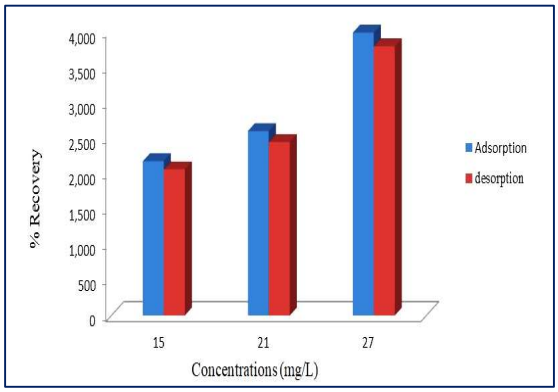


Fig.-5: Histogram of Percent Recovery of the Adsorption-Desorption Process of MIP_DEHP Using Several Concentration Variations

The histogram in Fig.-5 shows that almost all DEHP concentrations adsorbed by MIP can be desorbed using 100% methanol as solvent. The percent recovery from the three concentrations used was 94.66%, 94.40%, and 95.52%, respectively. This matter shows that MIP is possible to be reused as an adsorbent.

CONCLUSION

The functional materials of MIP_DEHP and NIP have been synthesized utilizing the precipitation polymerization method. The material is a white fine solid. The results of SEM characterization exhibited the surface morphology of MIP_DEHP in the form of granules that tend to be uniform in size with more delicate texture than NIP. The characterization results using TGA exhibited that MIP was stable at high temperatures. The adsorption kinetics model for MIP_DEHP$_{(AE)}$ was PSO. The adsorption capacity value of MIP_DEHP$_{(AE)}$ obtained from the isothermal adsorption model of Langmuir was 4.392 mg/g. Applying the synthesized MIP as an adsorbent through several stages of the separation process carried out in the SPE method, then MIP_DEHP proved to be very good for use as an adsorbent. MIP allows it to be reused as an adsorbent because a very high recovery percentage is obtained.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID IDs, given below:

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